PROPERTIES OF ABLATION AND INSULATION MATERIALS

Volume II

Thermal Properties of Three Rigidized Fibrous Insulators

by E. D. Smyly and C. D. Pears

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ABSTRACT

Thermal conductivity, thermal expansion and heat capacity were determined on three rigidized fibrous insulators. The materials consisted of either silica or similar type fibers rigidized with a binder. Properties were measured from 150 K to either 1900 K or the temperature at which severe shrinkage began. Thermal conductivity measurements were made in vacuum, air and nitrogen.

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SUMMARY

Thermal conductivity, enthalpy, thermal expansion and bulk density were measured on three rigidized fibrous insulations. The materials consisted of silica-like fibers with a binder which gave them rigidity. The materials were designated alumina-silica-chromia, mullite and LI-1500 rigidized insulators. The thermal conductivity in nitrogen was measured from 150 K to about 1300 K. In vacuum, thermal conductivity was measured from about 700 K to 1300 K. Enthalpy was measured from 150 K to 1900 K. Thermal expansion was measured from 150 K to about 1300-1500 K.

A thermal model was applied to the thermal conductivity data to indicate the mechanisms of heat flow through the materials and to provide a means of extrapolating the values for vacuum, within reasonable limits, outside the range of the measurements.

The mullite fiber and alumina-silica-chromia rigidized insulations had about the same expansion characteristics and similar thermal conductivity values in nitrogen. The LI-1500 had the lowest expansion and the lowest thermal conductivity in nitrogen. In vacuum, the mullite had the highest value for thermal conductivity and the LI-1500 the lowest.

All of the materials had the same heat capacity within about 5 percent. The heat capacities were similar to the values for fused silica.

All three materials exhibited significant shrinkage at elevated temperatures. The shrinkage began for the alumina-silica-chromia and LI-1500 at about 1300 K and for the mullite at about 1500 K. For the mullite, heat treatment at 1865 K did not alter the shrinkage behavior for a subsequent exposure.

INTRODUCTION

This is the final report to the National Aeronautics and Space Administration, Langley Research Center, for the work on rigidized insulators under Subtask A of Contract NAS1-7732-1. This work involved duplicate measurements of thermal conductivity, enthalpy (heat capacity was a derived quantity) and thermal expansion of three rigidized insulators from 150 K to 1900 K. Thermal conductivity measurements were made in air from 150 K to about 600 K with a guarded hot plate apparatus and in vacuum and nitrogen from about 700 K to 1300 K (mean temperature) with a radial inflow apparatus. Severe shrinkage of the materials above about 1300 K precluded thermal conductivity measurements to higher temperatures.

The three rigidized fibrous insulators on which measurements were made were alumina-silica-chromia, mullite and LI-1500.

MATERIALS AND CUTTING PLANS

The materials evaluated in this program were:

1. Alumina-silica-chromia rigidized insulation
2. Mullite fiber rigidized insulation
3. LI-1500

Pictures of the three materials are presented in Figure 1 and a limited amount of information is given in Table 1. The compositions of the materials were proprietary.

The mullite fiber and the LI-1500 were white in color and the alumina-silica-chromia was light green. Under a stereo microscope at about 30X the alumina-silica-chromia and mullite fiber appeared as small glass-like fibers with much void space between fibers. There were also spherical inclusions in these two materials but these were dispersed at large spacings. The binder was not readily apparent and was probably concentrated at the junctions of fibers. The LI-1500 was similar to the other two materials except that the voids were filled with a white powder and one could not see "into" the material. How well the powder adhered to the fibers is not known but it could be rubbed from the surface with some ease.

The alumina-silica-chromia and mullite were received in the form of squares about 10.12 cm square by 3.18 cm thick. The LI-1500 was received in the form of a block about 15.23 cm square by 6.85 cm thick. One square surface of the LI-1500 had a thin green layer. The thickness direction of the blocks was called the across lamina direction and the direction perpendicular to the thickness was called the with lamina direction. Specimens were obtained such that thermal conductivity was measured in the across lamina direction and thermal expansion was measured in both directions.

Ten blocks each of the alumina-silica-chromia and mullite were received. The values of bulk density for these blocks in the "as-received" condition are given in Tables 2 and 3. Also, the bulk density of the block of LI-1500 is given in Table 2. The densities of the blocks of the alumina-silica-chromia ranged from 0.216 gm/cm^3 to 0.264 gm/cm^3. The range of density for the mullite was 0.226 gm/cm^3 to 0.255 gm/cm^3. The density of the LI-1500 was 0.277 gm/cm^3. In addition to the density variations between blocks, density measurements made on specimens indicated some variations in density within the blocks of material.

The cutting plans used to obtain specimens from the materials are given in Figures 2, 3 and 4. The following code was used to identify specimens (except for those used for thermal conductivity measurements in the radial inflow apparatus):

```
AL - ASTM - 1C - W - 1
|     |      |   |   |___ Specimen number
|     |      |   |_______ Orientation
|     |      |___________ Block identification
|     |__________________ Property
|________________________ Material
```

AL - alumina-silica-chromia
MUL - mullite
LI-1500 - LI-1500
ASTM - thermal conductivity - ASTM C177 guarded hot plate
QEXP - thermal expansion - quartz dilatometer
GEXP - thermal expansion - graphite dilatometer
CPA - enthalpy - adiabatic calorimeter
CPI - enthalpy - ice calorimeter
W - with lamina
A - across lamina

APPARATUSES AND PROCEDURES

Thermal Conductivity

ASTM C177 Guarded Hot Plate. - This apparatus is described in Volume I. For this program, both the 3" and 7" apparatuses were used for measurements to 600 K. The specimen configuration for the 3" apparatus is given in Figure 5. For the 7" apparatus, a specimen 18.40 cm diameter by 0.635 cm thick was used. The method used to make the larger specimen is shown in Figure 6. The 2.54 cm thick blocks of material were sliced into three sections and then cut and fitted together such as to provide a size larger than that required for the specimen. The sections were joined together with a thin joint of silicone rubber (Siliastic® RTV 731). The specimens were then surfaced and cut to the proper diameter.

All thermal conductivity evaluations with the ASTM C177 guarded hot plate were made in air.

Radial Inflow Apparatus. - A radial inflow apparatus was used for thermal conductivity measurements from about 700 K to 1300 K. The basic apparatus for the measurements is described in Reference 1 and in Appendix A. For this program, a strip specimen technique was used. Two furnaces were used for the measurements. Both furnaces are similar to the high temperature furnace described in Reference 1. One furnace (load furnace) has been modified such that a positive compaction pressure can be applied to a strip specimen during a thermal conductivity evaluation. This furnace can be operated at about 1 atmosphere of pressure. The other furnace (GFE) is described in Appendix B. The GFE furnace can be operated at pressures ranging from less than 10 torr to 1380 $\times 10^3$ N/m^2 (200 psig). The GFE furnace was used for all of the elevated temperature measurements except one which was made in the load furnace. Basically, the radial inflow apparatus consists of a graphite heater tube which surrounds a specimen mounted on a water flow calorimeter centered axially through the specimen. Heat flows inwardly through the specimen into the calorimeter.

For the evaluations in this program, a strip specimen configuration was employed. A strip specimen configuration was selected for two reasons: (1) heat flow was desired in a specific direction, and (2) a thin specimen was desired so that maximum heat flow could be obtained. The strip configuration employed is shown in Figure 7. The specimen consisted of four strips arranged in a square array about the calorimeter. The dimensions of the specimen strips are shown in Figure 8. The strips were placed between temperature wells made of ATJ graphite. Note in Figure 7 that temperature measurements are made on both sides of each strip. Grafoil sheets about 0.013 cm thick were placed in the interfaces between the specimen strips and the temperature wells. The Grafoil was used to provide good interface contact. Experience has shown that Grafoil provides a high contact conductance, even in vacuum. Pyrolytic graphite strips 0.157 cm thick were used on the inside of the buildup. The large anisotropy in thermal conductivity of the pyrolytic graphite provides essentially flat isotherms perpendicular to the direction of heat flow through the strips. Pictures of the apparatus are shown in Figure 9.

For the one evaluation in the load furnace with positive compaction pressure control (Specimen AL-1), Grafoil was not used at the interface between the ATJ strips and the specimen. This evaluation was made to provide baseline data for comparison with the method used for the measurements in the GFE furnace. In this run, the specimen strips employed were 1.27 cm (0.5 inch) wide by 7.11 cm (2.8 inches) long.

Since temperatures are not measured directly in the specimen, the maintainence of good mechanical and thermal contact between the specimen and temperature wells was mandatory. Generally, this is not an unsolvable problem because most materials exhibit sufficient expansion to maintain hard contact with the temperature wells. However, the materials in this program had low expansions in the across lamina direction and shrunk at higher temperatures. Hence, some means were required to insure positive physical contact between the specimen and the ATJ strips. The means used to insure good interface contact are shown in Figure 7. Pistons of pyrolytic graphite were placed in graphite cylinders screwed into the support cylinder. Pictures of the PG pistons and holders are shown in Figure 9. The differential expansion between the pyrolytic graphite pistons and the ATJ support cylinder forced the outer temperature well against the specimen. From the dimensions given in Figure 7 and the known expansion curves for ATJ and PG, the decrease in the thickness of the gap between the temperature wells was calculated. The calculations were performed for gradients typical of those measured during a thermal conductivity evaluation. The results of the calculations are presented in Figure 10. Since the specimen materials were "soft" relative to the other components in the system, the specimens absorbed the strains and deformed as shown in Figure 10. Note that this method allows one to accomodate specimen shrinkages of up to 50 x 10^{-3} cm/cm at a face temperature of 1370 K for a 0.254 cm thick specimen. If the pistons are not used, the gap between the ATJ strips increases as a function of temperature.

The basic assumption for the configuration employing the strip specimen is that the heat flow divides equally between the four strips. Then the thermal conductivity of the specimen can be calculated from the following equation

$$K = \frac{\Delta X}{4A} \frac{Q}{\Delta T} \qquad (1)$$

where

K = thermal conductivity of specimen
A = gage area of one specimen strip (width times 1.27 cm axial gage length of calorimeter)
ΔT = temperature difference across specimen strip
ΔX = gage thickness of specimen
Q = total metered heat flow

The gage thickness normally used in the calculations was the initial thickness of the specimen strips minus the decrease in thickness as obtained from Figure 10.

In performing the thermal conductivity calculations, the temperature drops across the Grafoil and ATJ strips were calculated and subtracted from the total measured temperature difference to obtain the true temperature difference. The values of thermal conductivity used for the Grafoil are given in Figure 11 and the values of thermal conductivity for the ATJ are given in Appendix A. The corrections were typically less than 5 percent of the total temperature difference.

The evaluations with the radial inflow apparatus were made in the environmental furnace described in Appendix B. This furnace had feedthroughs for thermocouples and optical sight ports for access for viewing down into the temperature wells (shown in Figure 7) and onto the face of the specimen. Sapphire windows are used to cover the optical sight ports for measurements in vacuum. When viewing through the sight ports a correction was made for the attenuation by the windows.

Chromel/Alumel thermocouples were used for temperature measurements to about 1470 K. Above 1470 K, an optical pyrometer of the disappearing filament type was used. At 1470 K, both procedures have been used and the results have been in excellent agreement.

The mean temperature, with the strip configuration, is taken as the average of the hot and cold temperature readings when thermocouples are employed. When direct optical sightings down the thermocouple wells are used, the observed temperatures are not true temperatures. In this case, the mean temperature is calculated from the true face temperature by assuming a linear temperature gradient and using the following equation

$$T_{mean} = T_{of} - \frac{\Delta T}{2} \tag{2}$$

where

T_{mean} = mean temperature for the measurement
T_{of} = outer face temperature
ΔT = temperature difference across specimen

The basic uncertainty with the radial inflow apparatus on "well-behaved" materials is ±7 percent. For the particular measurements in this program, with the strip specimen employed, the random uncertainty is estimated to be ±10 percent. In addition to the random uncertainty, there is a systematic uncertainty due to the heat flow through the thermatomic carbon at the corners of the strip buildup. The systematic uncertainty is estimated by calculation to be no more than +10 percent. Hence, the total uncertainty is estimated as -10 percent to +20 percent (plus denotes the measured value is higher than the true value).

For the one evaluation in the load furnace (Specimen AL-1), a compaction pressure of 69×10^3 N/m^2 (10 psi) was employed and nitrogen was used as the purge gas. For the evaluations in the GFE furnace, data were obtained in vacuum and in nitrogen at atmospheric pressure. The vacuum level was less than 10 torr.

Enthalpy (Heat Capacity)

Adiabatic Calorimeter. - An adiabatic calorimeter was used for measurements of enthalpy from 150 K to 810 K. This apparatus is described in Volume I.

Ice Calorimeter. - An ice calorimeter was used for enthalpy measurements from 810 K to 1900 K. This apparatus is fully described in Appendix C.

The ice calorimeter requires a specimen 1.91 cm diameter by 1.91 cm long. Because of the low densities of the materials in this program, this specimen size would not provide sufficient weight to yield a signal as large as desired. The specimen weights were increased by pulverizing the materials and placing them in thin walled drop cups of the proper dimensions. The drop cups and covering lids were made from CS graphite and have been calibrated to provide the quantity of ice melted by the cup as a function of temperature. The pulverization provided more than twice the specimen weight in the same volume. The lid was held in place on the drop cup with a negligible quantity of graphite glue.

In performing the measurements, the total volume of ice melted by a drop into the calorimeter at a given specimen temperature is obtained. From this total signal, the volume of ice melted by the drop cup is subtracted to obtain the volume of ice melted by the specimen.

The specimens were heated in the high temperature furnaces in a helium environment. Measurements were made which insured that holding the powdered samples at temperature for 15 minutes provided a uniform temperature throughout.

Thermal Expansion

Quartz Dilatometer. - The quartz dilatometer which was employed for thermal expansion measurements to about 1100 K is described in Volume I.

Graphite Dilatometer. - A graphite dilatometer was used for thermal expansion measurements from 300 K to 1900 K. This apparatus employs a dilatometer tube and pushrod made from CS graphite. A dial gage is used to read relative motion between the specimen and dilatometer tube. The graphite dilatometer is fully described in Appendix D.

The specimen configuration for the measurements with the quartz and graphite dilatometers was 1.27 cm diameter by 7.61 cm long with a 7.61 cm radius on both ends. The diameter of the pushrod for the graphite dilatometer is 0.635 cm. If the pushrod achieves full contact with the specimen, the resulting contact pressure due to the weight of the components and the spring load in the dial gage is about 69×10^3 N/m^2 (10 psi). The stress in the main body of the specimen is about 17×10^3 N/m^2 (2.5 psi).

The graphite dilatometer was used in the high temperature furnace described in Reference 1. A helium purge was used for all evaluations.

Bulk Density

The bulk densities of all specimens were measured by the methods described in Volume I.

Equation for Analysis of Thermal Conductivity Data

A superposition model was used to analyze the thermal conductivity data. Such a model is probably not rigorous for these materials but a more detailed model would require extensive material characterization and analysis.

The superposition model assumes parallel heat flows by gas conduction, solid conduction and radiation. Convection heat transfer was assumed to be negligible. The equation used was

$$k_{eff} = (1-P^{2/3})k_m + P^{2/3}k_g + \frac{4\sigma T^3}{N} \qquad (3)$$

where

k_{eff} = effective thermal conductivity
P = porosity
k_m = thermal conductivity of solid
k_g = thermal conductivity of gas
σ = Stefan-Boltzmann constant
T = absolute temperature
N = backscattering cross section per unit volume

In Equation 3, $(1-P^{2/3})k_m$ represents the contribution to the effective conductivity due to the solid conduction. The use of the 2/3 power is an approximation and is discussed in Reference 1. The radiation term in Equation 3, $4\sigma T^3/N$, is also discussed in Reference 1 and is a valid approximation for materials which attenuate radiation primarily by scattering and which have sufficient thickness. It is believed that these glass-like materials do attenuate radiation by scattering at air-fiber interfaces.

In using Equation 3, $(1-P^{2/3})\ k_m$ was estimated by subtracting the gas contribution $(P^{2/3}kg)$ from the value measured in nitrogen at 400 K. The backscattering parameter N, was determined from the effective value of thermal conductivity measured in vacuum at 1200 K by subtracting the solid conduction component. Then, using the reduced values, thermal conductivity values were predicted at various temperatures for environments of vacuum and nitrogen and compared with the measured values.

In the analysis, the solid conduction component of thermal conductivity and the backscattering cross section per unit volume were assumed constant. The solid conduction component probably changes with temperature; however, the variation is not defined for these materials. If the change is 20 percent or less the results would be affected at most by 0.008 W/m-K. The backscattering cross section may change with temperature; however, studies by another investigator (Reference 2) led to the conclusion that the effect of temperature on absorption and scattering parameters was relatively small for the insulation materials which he studied, some of which were similar in structure and properties to those evaluated in this program. Hence, the assumption of constancy of the backscattering cross section seems reasonable.

The "true" solid conductivity was also estimated by dividing the solid conduction component by $(1-P^{2/3})$. This value is probably not rigorous for the simplified analysis used.

The values of porosity used in the analysis were estimates only. Porosity was estimated by assuming a true density of 2 gm/cm^3 and using the measured values of bulk density.

The analysis was performed to provide some indication of the character of the vacuum data and to allow some extrapolation since data were obtained only over a limited temperature interval.

DATA AND RESULTS

Thermal Conductivity

Alumina-Silica-Chromia. - The thermal conductivity data for the alumina-silica-chromia are presented in Tables 4 through 8 and in Figures 12 and 13. Shown in Figure 12 are the data obtained with the guarded hot plate apparatus. Note that Specimen AL-ASTM-3C-2 and 3D-2 had the higher density and also the higher thermal conductivity. All of the data are plotted in Figure 13. In nitrogen, the thermal conductivity increased from 0.042 W/m-K at 150 K to 0.267 W/m-K at 1300 K. In vacuum the thermal conductivity increased from about 0.06 W/m-K at 700 K to 0.15 W/m-K at 1300 K.

One specimen (AL-1) was evaluated with the radial inflow apparatus in the load furnace at a positive compaction pressure of $69 \times 10^3\ N/m^2$ (10 psi). The data for this specimen were higher than those of the other specimens at low temperatures but converged to about the same value at 1200 K.

Mullite. - The thermal conductivity data for the mullite fiber rigidized insulation are presented in Tables 9 through 12 and in Figures 14 and 15. Shown in Figure 14 are the data obtained in air with the ASTM C177 guarded hot plate. The specimen evaluated in the 7 inch apparatus (MUL-ASTM-3B-3C) had the lower compaction pressure during the measurements and the higher thermal conductivity. However, it also had the higher density. All of the thermal conductivity data are presented in Figure 15. There was considerable scatter between the two specimens evaluated in the radial inflow apparatus just as there was between the two specimens evaluated with the guarded hot plate. The differences did not correlate well with density. A range of values has been given in Figure 15. Without additional study the causes for the range of values cannot be determined and one must consider the variations in using the data.

In nitrogen, based on the best curve through the data in Figure 15, the thermal conductivity increased from 0.073 W/m-K at 400 K to 0.27 W/m-K at 1300 K. In vacuum, the thermal conductivity increased from 0.06 W/m-K at 700 K to 0.19 W/m-K at 1300 K.

LI-1500. - The thermal conductivity data for the LI-1500 are presented in Tables 13 through 16 and in Figures 16 and 17. For the LI-1500, there was consistency between duplicate specimens. As shown in Figure 17, the data for the ASTM C177 and radial inflow apparatuses did not match too well at 600-700 K. However, this temperature range represents end points on both apparatuses and the smooth curve drawn through the data is considered representative of the true thermal conductivity of the material.

In nitrogen, the thermal conductivity of the LI-1500 increased from 0.055 W/m-K at 400 K to 0.21 W/m-K at 1300 K. In vacuum, the thermal conductivity increased from 0.04 W/m-K at 700 K to 0.08 W/m-K at 1300 K.

Enthalpy

Alumina-Silica-Chromia. - The enthalpy data for the alumina-silica-chromia are presented in Tables 17 and 18 and in Figure 18. The enthalpy increased smoothly to a value of 19.4 x 10^5 J/Kg at 1900 K. The heat capacity is given in the upper portion of Figure 18. There was good agreement between the analytical and graphical solutions for heat capacity. The heat capacity increased smoothly from 600 J/Kg-K at 200 K to 1470 J/Kg-K at 1900 K.

Mullite. - The enthalpy data for the mullite are presented in Tables 19 and 20 and in Figure 19. The enthalpy increased smoothly to a value of 19.3 x 10^5 J/Kg at 1900 K. The heat capacity is shown in the upper portion of Figure 19. There was some disagreement between analytical and graphical values of heat capacity primarily because of the differences in the analytical and hand fits of the enthalpy data. A judgement was made and in our judgement the curves drawn represent the best values of enthalpy and heat capacity. The heat capacity increased from 610 J/Kg-K at 200 K to 1430 J/Kg-K at 1900 K.

LI-1500. - The enthalpy data for the LI-1500 are presented in Tables 21 and 22 and in Figure 20. The enthalpy increased smoothly to a value of 19×10^5 J/Kg at 1900 K. The heat capacity is given in the upper portion of Figure 20. The analytical values of heat capacity differed significantly from the graphically obtained values. The difference was primarily due to a poor analytical fit of the enthalpy data. This was probably caused by the low experimental data at 800 and 1000 K which were ignored in hand fitting the data. It is recommended that the heat capacity curve drawn in the figure be used and the analytically determined values of heat capacity be rejected. The heat capacity increased from about 600 J/Kg-K at 200 K to 1400 J/Kg-K at 1900 K.

Enthalpy Specimens. - Post-exposure pictures of the enthalpy specimens used in the ice calorimeter are presented in Figure 21. The specimens were initially powdered. Exposure to 1800-1900 K resulted in sintering of the powder into solid cylinders. The three materials behaved somewhat differently. The alumina-silica-chromia powder exhibited the most shrinkage and the mullite the least. The LI-1500 maintained its original white coloration. The alumina-silica-chromia took on a bluish tint. The mullite exhibited little shrinkage but took on a brownish tint due to the exposure. The color changes may be related to reactions between the materials and the carbon environment. However, the changes introduced no sudden shift in properties.

Thermal Expansion

Alumina-Silica-Chromia Rigidized Insulation. - Thermal expansion data for the alumina-silica-chromia are presented in Tables 23 through 27 and in Figures 22 and 23. The data for the thermal expansion in the with lamina direction are presented in Figure 22. There was a significant amount of scatter between specimens. The thermal expansion increased to a maximum value of about 3×10^{-3} cm/cm at 1100 K. Above 1300 K, the material exhibited a gross shrinkage. The permanent shrinkages of the specimens as a result of exposure, are summarized in Table 28. The maximum permanent shrinkage observed for the with lamina direction after exposure to 1497 K was 278×10^{-3} cm/cm. Recall that a load of about 69×10^3 N/m^2 (10 psi) was at the contact point and the uniform stress in the specimen was about 17×10^3 N/m^2 (2.5 psi).

In the across lamina direction (see Figure 23), the alumina-silica-chromia exhibited a higher expansion than in the with lamina direction, about 4.7×10^{-3} cm/cm at 830 K. However, in the across lamina direction, the material began to shrink at 830 K and exhibited a higher value of permanent shrinkage than in the with lamina direction (about 674×10^{-3} cm/cm).

One specimen of the alumina-silica-chromia was heat soaked to 1861 K with no load on the specimen (see Table 28). The permanent shrinkage of this specimen was 532×10^{-3} cm/cm.

Mullite Fiber Rigidized Insulation. - The thermal expansion data for the mullite fiber are presented in Tables 29 through 36 and in Figures 24 and 25. The data for the with lamina direction are presented in Figure 24. Of three specimens evaluated in the "as-received" condition with the graphite dilatometer, the data for two were in good agreement with the quartz dilatometer and the data for one fell much lower. In our judgement, the higher values represent the best values since there were four replications which were in good agreement. In the "as-received" condition, the thermal expansion in the with lamina direction increased to a maximum of about 5.7×10^{-3} cm/cm at 1500 K and exhibited severe shrinkage above this temperature.

The thermal expansions of two specimens were measured in the with lamina direction after a heat soak at 1865 K. These specimens had less expansion than the "as-received" material and reached a maximum of about 4×10^{-3} cm/cm at 1500 K. However, the specimens which had been heat soaked again exhibited a gross shrinkage beginning at 1500 K.

In the with lamina direction, permanent shrinkages of from 109×10^{-3} to 170×10^{-3} cm/cm were noted after thermal exposure (see Table 28). Likewise, the specimens heat soaked prior to the evaluation also exhibited gross permanent shrinkages of about 110×10^{-3} cm/cm. However, during the heat soak (prior to the expansion evaluation) the specimens were unloaded and permanently shrunk only 8×10^{-3} and 10×10^{-3} cm/cm after exposure to 1865 K.

The data for the thermal expansion of the mullite fiber in the across lamina direction are presented in Figure 25. In the across lamina direction the material expanded to a maximum value of about 4.2×10^{-3} cm/cm at 1200 K and exhibited gross shrinkage above 1500 K. The permanent shrinkage after exposure to 1697 K was 148×10^{-3} cm/cm.

LI-1500. - The data for the thermal expansion of the LI-1500 are presented in Tables 37 through 41 and in Figures 26 and 27. The data for the expansion in the with lamina direction are presented in Figure 26. This material exhibited a low value of expansion, reaching a maximum of 0.6×10^{-3} cm/cm at about 1050 K. Gross shrinkage was observed for the with lamina direction at temperatures above 1300 K. Permanent shrinkages of 83×10^{-3} and 84×10^{-3} cm/cm were observed after exposures to 1458 and 1883 K respectively. One specimen was heat soaked to 1864 K with no load on the specimen (see Table 28). The permanent shrinkage of this specimen after exposure to 1864 K was 45×10^{-3} cm/cm.

In the across lamina direction (see Figure 27), the behavior of the thermal expansion of the LI-1500 was similar to that for the with lamina direction. The expansion peaked at 0.75×10^{-3} cm/cm at 1100 K and had severe shrinkage above 1300 K. The permanent shrinkage in the across lamina direction after exposure to 1922 K was 158×10^{-3} cm/cm.

Pictures of Thermal Expansion Specimens. - Post-exposure pictures of the thermal expansion specimens are presented in Figure 21. From the pictures, the severe shrinkage of the alumina-silica-chromia is apparent. Note on the ends of the specimens that the pushrod indented the specimens. This indicates softening of the materials to a degree that a load of 69×10^3 N/m^2 (10 psi) could cause permanent deformation.

DISCUSSION

The thermal conductivities of the alumina-silica-chromia, mullite fiber and LI-1500 rigidized insulations are overplotted in Figures 28 and 29 for environments of nitrogen (air) and vacuum, respectively. In nitrogen, the thermal conductivities of the alumina-silica-chromia and mullite were very similar. The thermal conductivity of the LI-1500 was about 25 percent lower than that of the other two materials. Literature data (Reference 3) for similar rigidized fibrous insulators are presented in Figure 28 for comparison. The reference data were in general agreement in level and character with the values measured in this program.

In vacuum, the mullite fiber had the highest thermal conductivity followed in order by the alumina-silica-chromia and LI-1500 which were about 20 and 50 percent lower, respectively.

The results of the analysis of the thermal conductivity data are presented in Figures 30 through 32. The parameters used in the data analysis and the values reduced from the data with the use of Equation 3 are presented in Table 42. The data correlation for the alumina-silica-chromia is shown in Figure 30. Recall that the vacuum data were primarily used to obtain the radiation parameter. The experimental data grouped well about the analytical curve with a value of N, backscattering cross-section per unit volume, of 4900 m^{-1}. The values predicted for nitrogen fell about 19 percent below the experimental data.

The data correlation for the mullite fiber are presented in Figure 31. The experimental data grouped rather well about the analytical curve, but with a great deal of scatter. The value obtained for N for this material was 3160 m^{-1}. The thermal conductivity values predicted for nitrogen agreed well with the experimental data.

The data correlation for the LI-1500 is presented in Figure 32. The experimental data in vacuum grouped reasonably well about the analytical curve. The value calculated for N was 7130 m^{-1}. The thermal conductivity calculated for a nitrogen environment fell about 18 percent lower than the measured values.

The results of the analysis of the thermal conductivity data are summarized in Table 42. The values in the table were used in and obtained from the application of Equation 3 to the experimental data. The solid conduction contributions for the alumina-silica-chromia and mullite were about twice that of the LI-1500. The estimated values of "true" solid conductivity ranged from 0.29 to 0.66 W/m-K. For comparison, the thermal conductivity of slip cast fused silica is about 0.93 W/m-K at 400 K and increases with temperature. Hence, the level of solid thermal conductivity reduced from the data is of the right order.

The mullite fiber was the least effective in blocking radiant transport, as can be seen in Table 42 by observing that this material had the lowest value of N. Also shown in Table 42 is an estimate of the percent of the available heat flux being transmitted by radiation. One might think of this as being similar to transmittance. The LI-1500 transmitted about 5.5 percent of the available radiant energy and the mullite transmitted about 12.5 percent.

The fact that the LI-1500 had less radiant transport was probably related to the powder filler in the material.

The analytical curves for thermal conductivity agreed well with the experimental data in vacuum (primarily because the radiation parameter was obtained from these data). This provided a means of extrapolating the vacuum data somewhat outside the range of the measurements with some confidence. The analytical model (Equation 3) predicted reasonably well for a nitrogen environment but generally gave values which were too low. A model related more specifically to the materials would be required to provide a closer correlation of the experimental data under all conditions.

The heat capacities of all three of the materials were nearly the same. The heat capacities at 200 K were all about 600 J/Kg-K and increased smoothly to values at 1900 K ranging from 1400 J/Kg-K to 1470 J/Kg-K (a range of about 5 percent). Enthalpy values for fused silica are overplotted on the data for the alumina-silica-chromia in Figure 18. The enthalpy of the alumina-silica-chromia was about 10 percent higher than that of fused silica.

In the with lamina direction, the mullite fiber had the highest thermal expansion of the three materials and the LI-1500 had the lowest expansion. The relative maximum values were about 5.7×10^{-3} cm/cm for the mullite and 0.5×10^{-3} cm/cm for the LI-1500. The alumina-silica-chromia and the LI-1500 exhibited gross shrinkages beginning at about 1300 K and severe shrinkage of the mullite began at about 1500 K.

All of the materials exhibited severe permanent shrinkages after exposure to about 1500 K or above. For the mullite fiber and LI-1500 there was some evidence that the permanent deformation was related to the load on the specimens during the measurements. Note in Table 28 that the specimens of these materials which were heat soaked without an imposed load exhibited less permanent shrinkage. However, even in the unloaded condition, the alumina-silica-chromia exhibited a permanent deformation in the with lamina direction of 532×10^{-3} cm/cm after exposure to 1861 K. The alumina-silica-chromia and the LI-1500 had more permanent deformation in the across lamina direction than in the with lamina direction.

CONCLUDING REMARKS

The thermal conductivities of three rigidized fibrous insulators were measured in nitrogen from 150 K to 1300 K and in vacuum from 700 K to 1300 K. Severe material shrinkage precluded thermal conductivity measurements to 1900 K. The mullite fiber rigidized insulation had the highest thermal conductivity in vacuum followed in order by the alumina-silica-chromia rigidized insulation and the LI-1500. In nitrogen, the alumina-silica-chromia and mullite had about the same values of thermal conductivity. The LI-1500 had the lowest thermal conductivity in both environments.

The mullite fiber rigidized insulation exhibited the most scatter in thermal conductivity. More replications would be required to confirm this behavior and establish the variability and mean value with more confidence.

A thermal model was fitted to the experimental data (see Equation 3). This model was useful to provide a means of extrapolating the vacuum data, within reasonable limits, outside the range of the measured values. The model generally predicted low for a nitrogen environment (about 19 percent) and more material characterization would be required to refine the model.

Heat capacity was determined from 200 K to 1900 K on three rigidized fibrous insulators. All three materials had the same values of heat capacity within about 5 percent. The enthalpy values were similar to that of fused silica.

Thermal expansion was measured from 150 K to about 1300-1500 K. All of the materials exhibited an increasing expansion to about 1000-1200 K with the mullite having the highest expansion and the LI-1500 the lowest. All of the materials exhibited gross shrinkages beginning at about 1300-1500 K. This shrinkage could not be removed from the mullite by heat treatment to 1865 K. For the mullite, the shrinkage resulting from exposures above 1700 K seemed to be dependent upon the load imposed on the specimen. This was probably true to some extent for the other materials also, but they exhibited severe shrinkages even when unloaded during thermal exposure.

The heat capacities and thermal expansions of the three rigidized fibrous insulators have been determined within a reasonable uncertainty such that the values reported herein may be used with confidence. However more thermal conductivity replications would be required to establish variability and provide high confidence data for design purposes.

Southern Research Institute
Birmingham, Alabama
November, 1970

APPENDIX A

THERMAL CONDUCTIVITY TO 5500°F BY RADIAL INFLOW METHOD

The thermal conductivity is determined with a radial heat inflow apparatus that utilizes a central specimen 1" long. This apparatus is normally employed for measurements over the temperature range from 1500°F to 5500°F. A comparative rod apparatus is used at temperatures below 1500°F where radiant heating is less effective. The radial inflow apparatus gives a direct measurement of the thermal conductivity rather than a measurement relative to some standard reference material. A picture of the apparatus ready to be installed in the furnace is shown in Figure A1. The furnace and associated equipment for the thermal conductivity work is shown in Figure A2. In addition to the specimen, the apparatus consists primarily of (1) a water calorimeter that passes axially through the center of the specimen, (2) guards made from the same specimen material at both ends of the specimens to reduce axial heat losses, (3) sight tubes that allow the temperature at selected points in the specimen to be determined either by thermocouples or optical pyrometer, and (4) an external radiant heat source (see Figure A3). The water calorimeter provides a heat sink at the center of the specimen to create a substantial heat flow through the specimen and allows the absolute value of the heat flow to be determined. Thermocouples mounted 1/2" apart in the calorimeter water stream measure the temperature rise of the water as it passes through the gage portion of the specimen. By metering the water flow through the calorimeter, it is possible to calculate the total radial heat flow through the 1/2" gage section of the specimen from the standard relationship $Q = MC\Delta T$. M is the weight of water flowing per hour, C is the specific heat of water, and ΔT is the temperature rise of the water as it passes through the gage section.

The standard specimen configuration is shown in Figure A4. The specimen is 1.062" O.D. x 0.250" I.D. x 1" long. Holes 0.073" in diameter are drilled on radii of 0.233 and 0.437 inch to permit measurement of the radial temperature gradient. In specimens which are anisotropic in the diametral plane (for example, certain graphites) a second pair of holes is drilled 90° to the first pair. The diameters joining each pair of holes is located to coincide with the principal planes of anisotropy in the material.

A 1/2" long upper guard and a 1/2" long lower guard of specimen material are placed above and below the 1" long specimen to maintain a constant radial temperature gradient throughout the entire specimen length and thereby prevent axial heat flow in the specimen. The outer ends of the specimen guards are insulated with graphite tubes filled with thermatomic carbon. These tubes also hold the specimen in alignment. The combined effect of specimen guards and thermatomic carbon insulation permits a minimum axial temperature gradient within the specimen. This gradient is not detectable by optical pyrometer readings. Visual inspection of the specimens after runs have verified that no large axial temperature gradient exists in the specimen. The guards, made of specimen material, display axial distortion of the isothermal lines for approximately 1/4" from the outer ends before reaching an apparent constant axial temperature.

When sufficient material is available the alternate specimen configuration shown in Figure A5 is employed. This specimen, being 1.5" in diameter, provides a larger gage length (0.357") between temperature wells and allows the installation of three holes on each radius without excessively distorting the radial temperature profiles. Thus this specimen configuration permits a more precise measurement of the average temperature at each radial location. As with the smaller specimen, the location of the temperature wells must be altered for transversely anisotropic specimens.

The annulus between the specimen inside diameter and the 7/32" outside diameter of the calorimeter tube is packed with either copper granules, graphite or zirconia powder. This packing provides a positive method for centering the calorimeter within the specimen and promotes good heat transfer between specimen and calorimeter.

Temperatures up to 2000°F are measured with Chromel/Alumel thermocouples inserted into the specimen through the sight tubes. At high temperatures the temperatures are measured through the vertical sight tubes using a right-angle mirror device and optical pyrometer.

In Figures A1 and A3 showing a typical conductivity calorimeter apparatus ready for insertion into a furnace for a run, a water-cooled copper section can be seen at the top of the unit. This section provides permanent sight tubes to within about 2-1/2" of the guard specimen, in addition to a permanent mount for the right-angle mirror device used with the optical pyrometer. Within the short zone between the water-cooled section and the top guard, thin-walled graphite sight tubes are fitted. The remainder of the annulus is filled with thermatomic carbon insulation.

During thermal conductivity runs, the following data are recorded: (1) power input, (2) specimen face temperature, (3) specimen temperatures in the gage section at the two radii, (4) temperature of the calorimeter water at two points 1/2" apart axially within the specimen center, and (5) water flow rate through the calorimeter. At least 5 readings are made at each general temperature range to determine the normal data scatter and to minimize the error that might be encountered in a single reading.

All thermocouple readings are measured on a Leeds and Northrup K-3 null balance potentiometer used in conjunction with a galvanometer of 0.43 microvolts per mm deflection sensitivity. All optically measured temperatures are read with a Leeds and Northrup Type 8622 optical pyrometer. The flow rate of the calorimeter water is measured with a Fischer and Porter Stabl-Vis Flowrater.

The thermal conductivity values are computed from the relation

$$K = \frac{QL}{\Delta T A_{1m}}$$

where Q is the heat flow to the calorimeter within the specimen gage section, A_{1m} is the log mean area for the specimen gage length, ΔT is the specimen temperature change across the specimen gage length, and L is the gage length over which the specimen ΔT is measured.

The heat flow Q is determined by the calorimeter. A_{1m} and L are calculated directly for the particular specimen configuration. ΔT is determined directly from the observed temperature difference across the specimen gage length.

Based on an extensive error analysis and calibrations on homogeneous isotropic materials of known thermal conductivities, such as Armco Iron and tungsten, the precision (coefficient of variation) in the measurements has been established at ±7 percent over the temperature range. For multiple runs on samples having similar properties, the uncertainty in a smooth curve through the data can be established to within ±7 percent. A detailed error analysis has been presented in a paper by Mann and Pears.[1]

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[1]Mann, W. H. Jr., and Pears, C. D. "A Radial Heat Flow Method for the Measurement of Thermal Conductivity to 5200°F", presented at the Conference on Thermal Conductivity Methods, Battelle Memorial Institute, October 26-28, 1961.

Data obtained here on several high temperature materials are presented in Figures A6, A7 and A8. Figure A6 is a plot of data obtained here on tungsten. The specimen for these determinations were fabricated from stacks of 0.060 inch washers cut from hot rolled sheet stock. Also plotted are values reported by other investigators including "recommended values" given by Powell, Ho and Liley[2] based on a compilation of 103 sets of data. Agreement between our data and the recommended values is excellent throughout the temperature range.

Figure A7 shows data obtained here on ATJ graphite, with grain. This material is premium grade, medium grain graphite having a density range of 1.73 to 1.78 gm/cm^3. The crosses (+) shown in the figure are "recommended values"given by Ho, Powell and Liley.[3] Again agreement is excellent.

Figure A8 shows data obtained on AXM-5Q1. These data were obtained under a program sponsored by the Air Force Materials Laboratory to develop high temperature thermal conductivity standards. Measurements were made on this material by four laboratories in addition to Southern Research Insitute. The bands shown in Figure A8 represent the range of data reported by the other participating organizations. A complete presentation and discussion of the data is given in AFML-TR-69-2[4].

[2] Powell, R. W., Ho, C. Y. and Liley, P. E. "Thermal Conductivity of Selected Materials" NSRDS-NBS 8, National Standard Reference Data Series - National Bureau of Standard - 8, 1966, pp. 11, 54-59.

[3] Powell, R. W., Ho, C. Y. and Liley, P. E. "Thermal Conductivity of Selected Materials, Part 2" NSRDS-NBS 16, National Standard Reference Data Series - National Bureau of Standards - 16, 1968, pp. 89-128.

[4] AFML-TR-69-2, "Development of High Temperature Thermal Conductivity Standards" submitted by Arthur D. Little, Inc., under contract AF33(615)-2874, 1969, pp. 115-127.

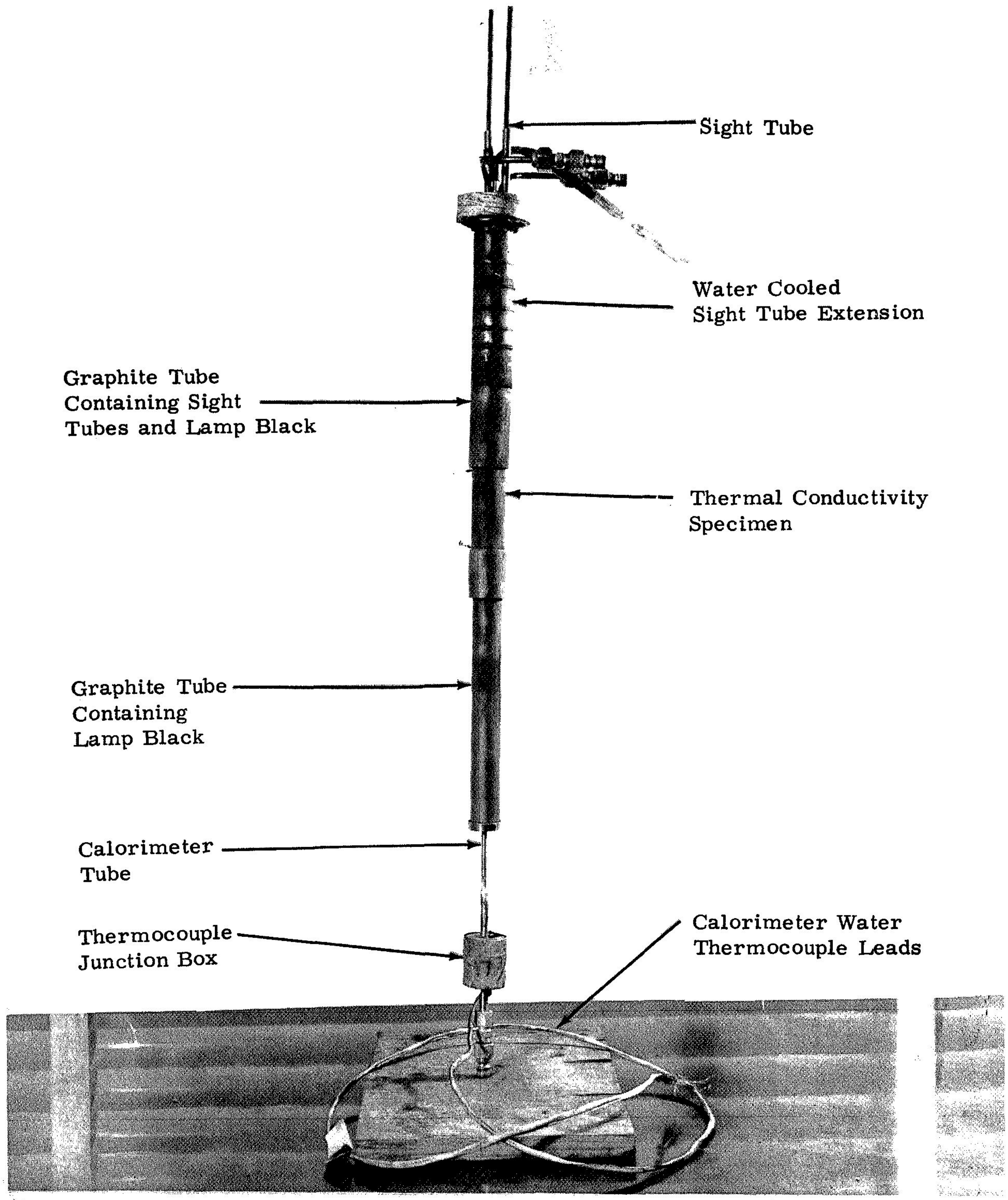

Figure A1. Picture of the radial thermal conductivity apparatus

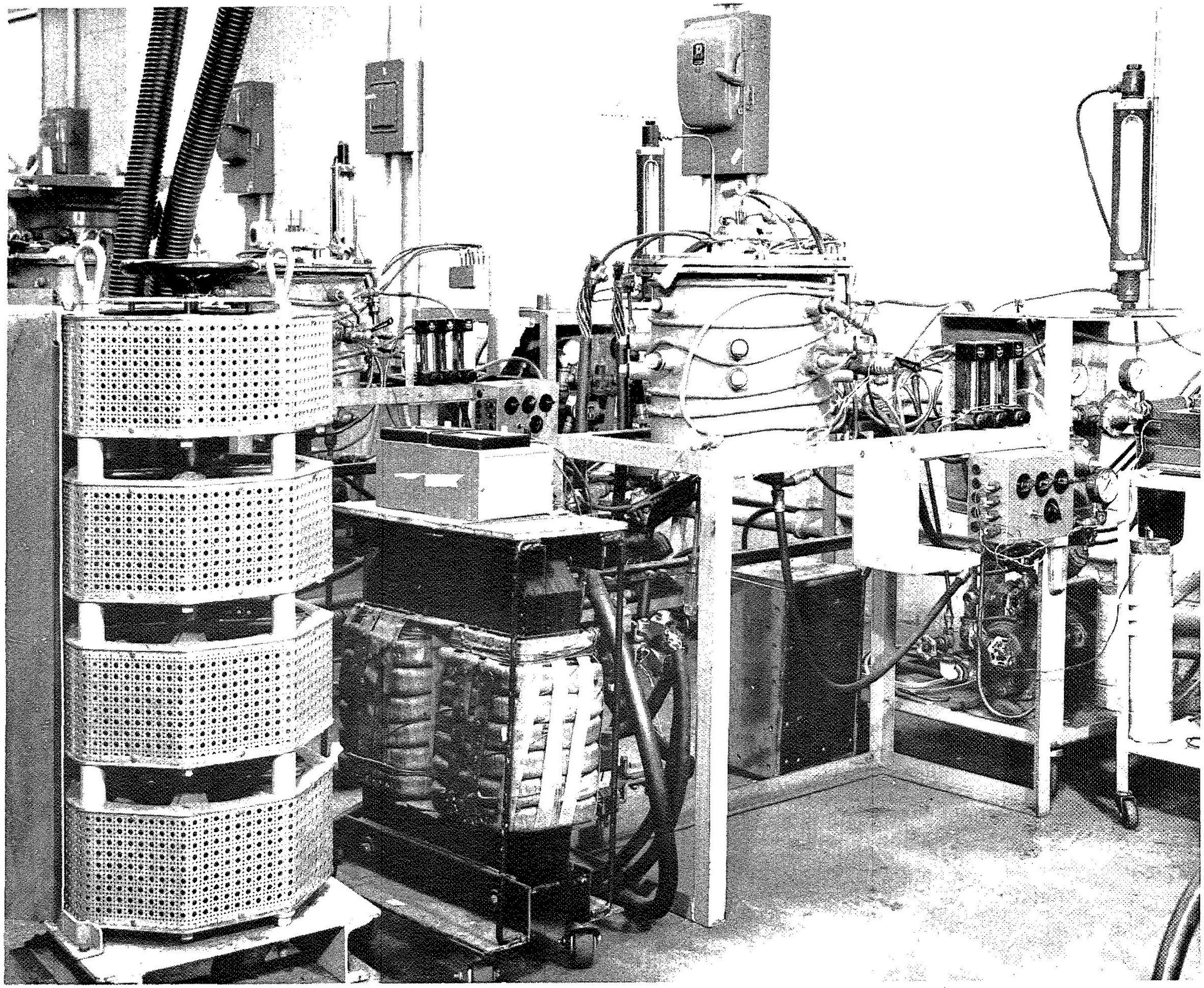

Figure A2. Furnace with thermal conductivity apparatus installed

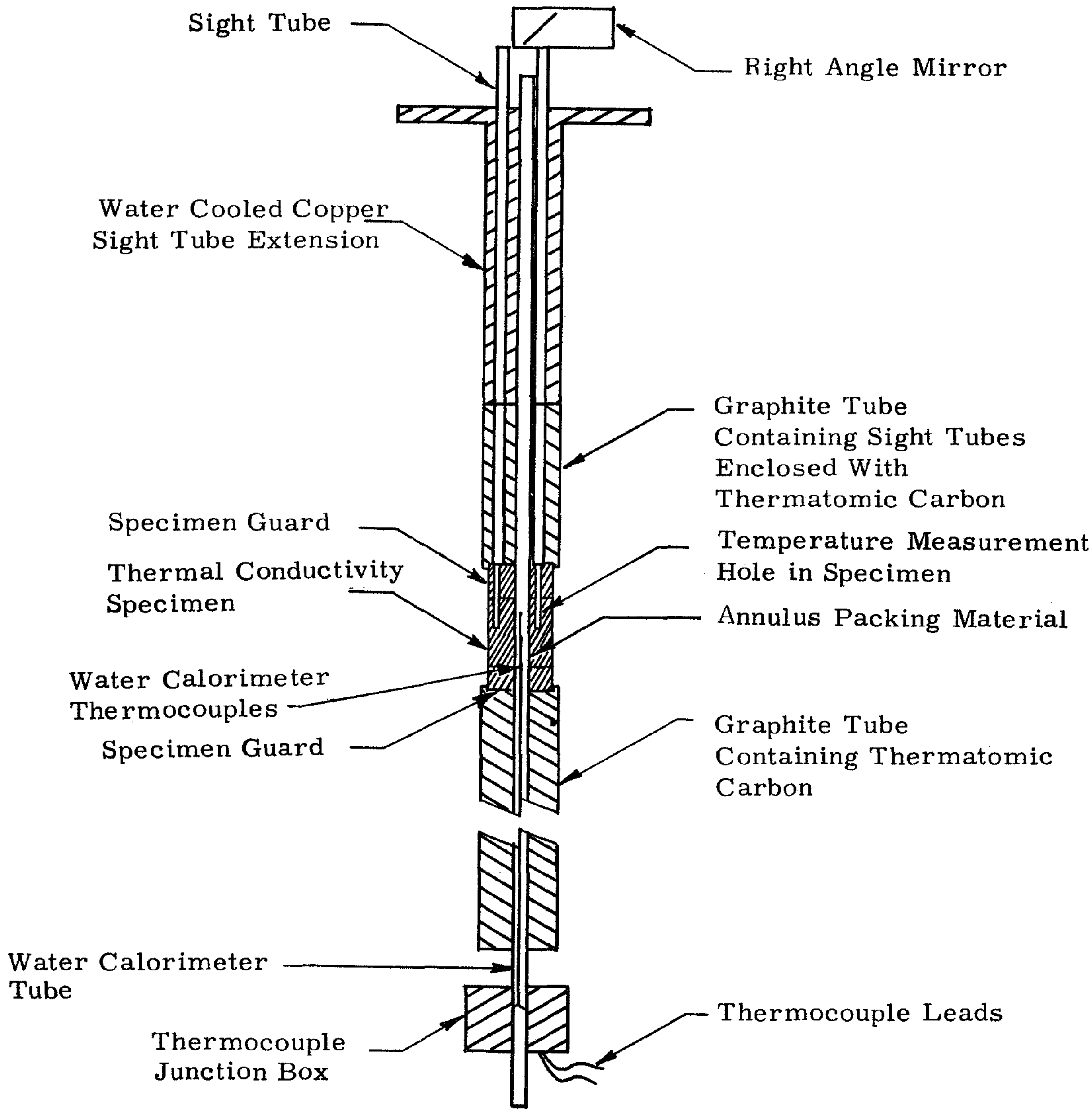

Figure A3. Cross-section of the thermal conductivity apparatus

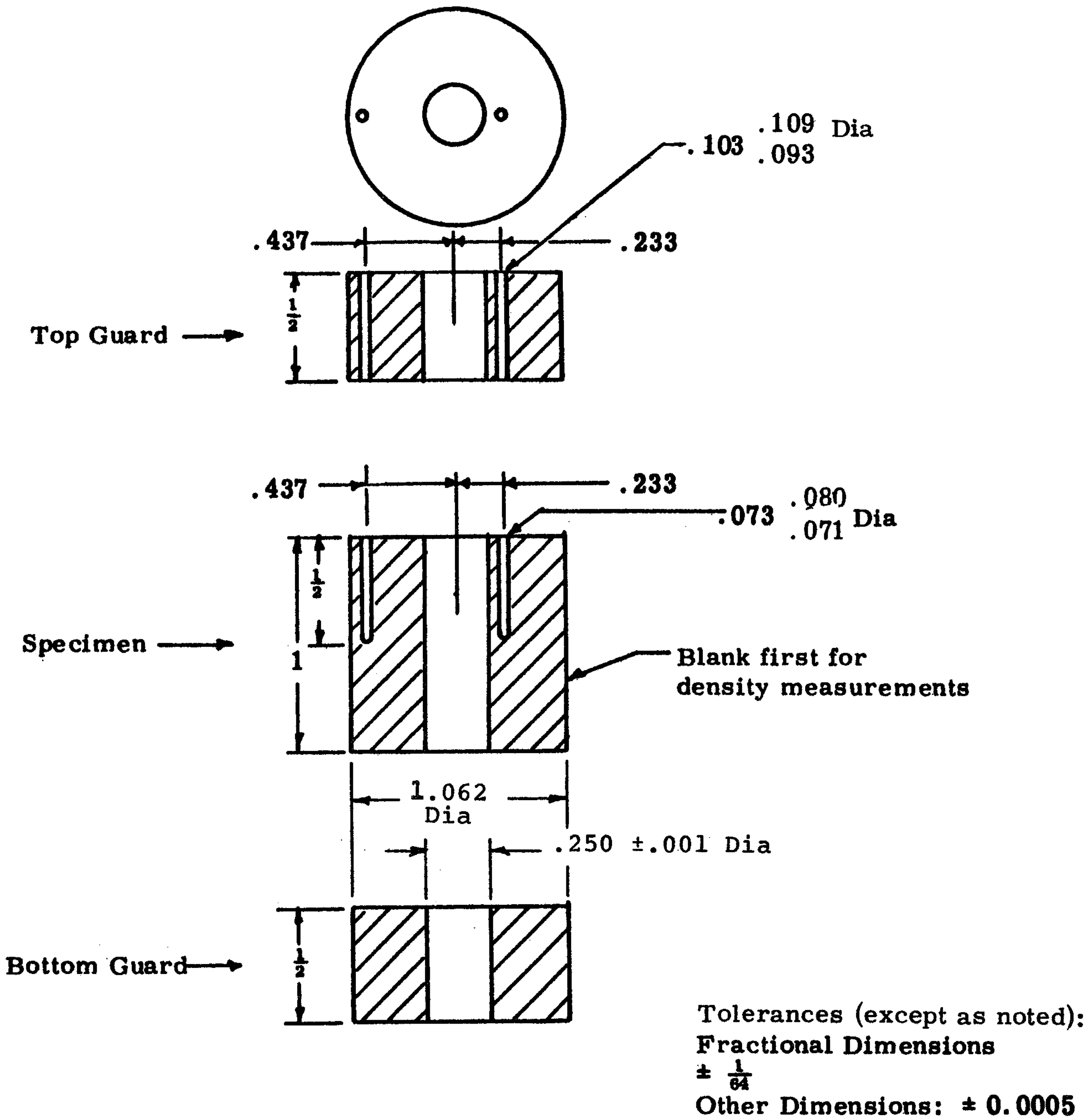

Figure A4. 1.06 Diameter thermal conductivity specimen for radial inflow apparatus

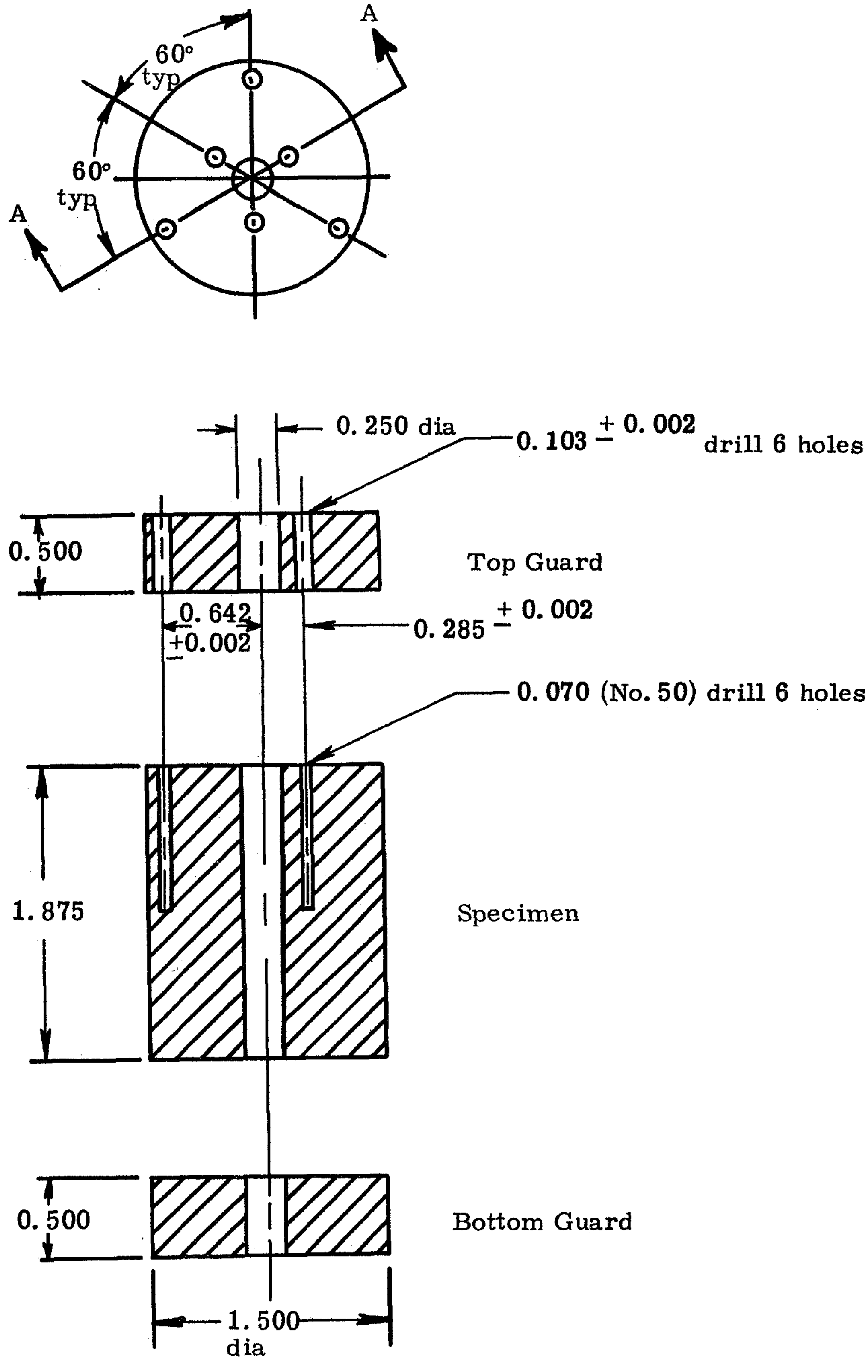

Figure A5. Dimensions of 1.50 inch diameter specimen used for radial inflow thermal conductivity measurements

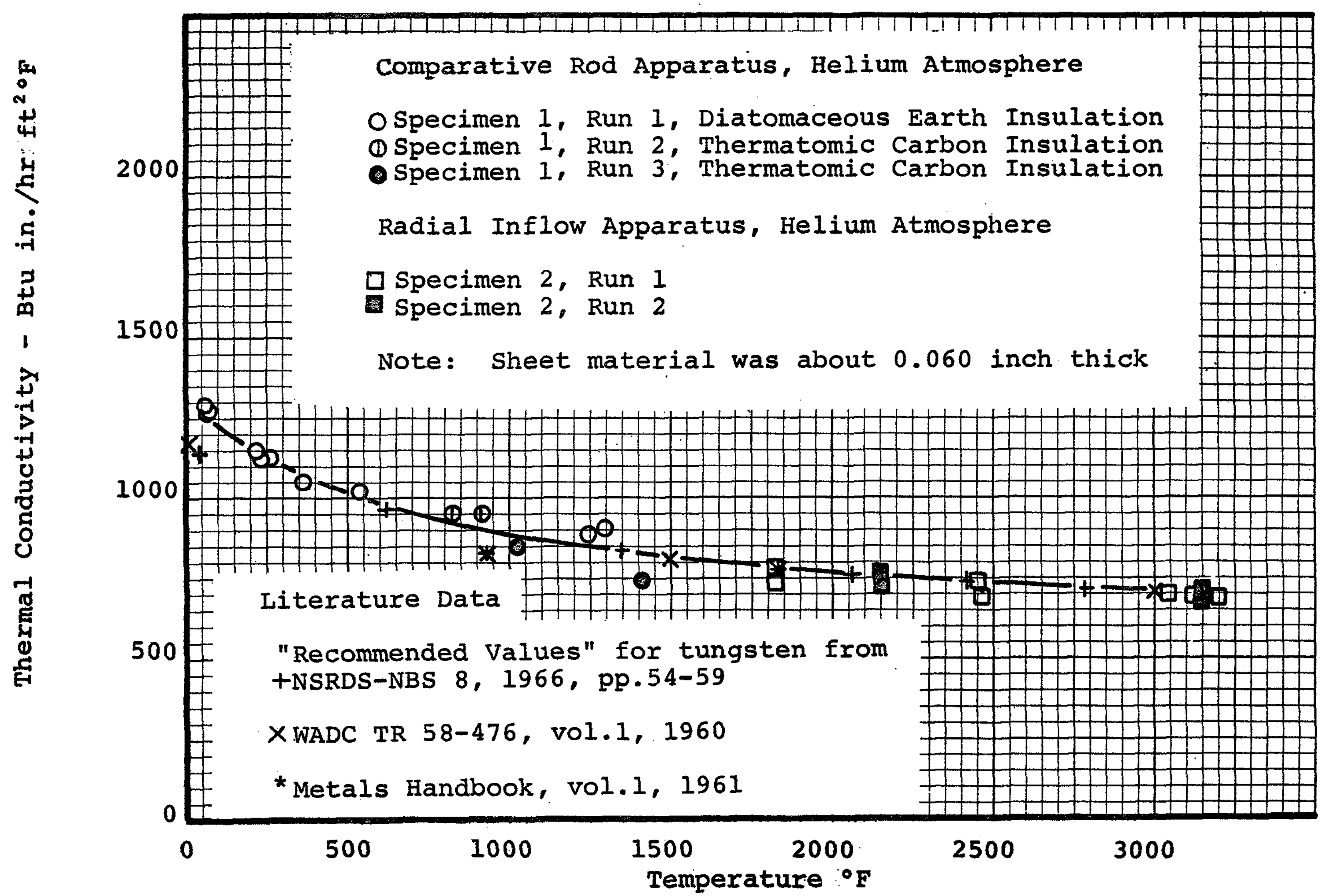

Figure A6. Thermal conductivity of tungsten sheet parallel to the plane of the sheet

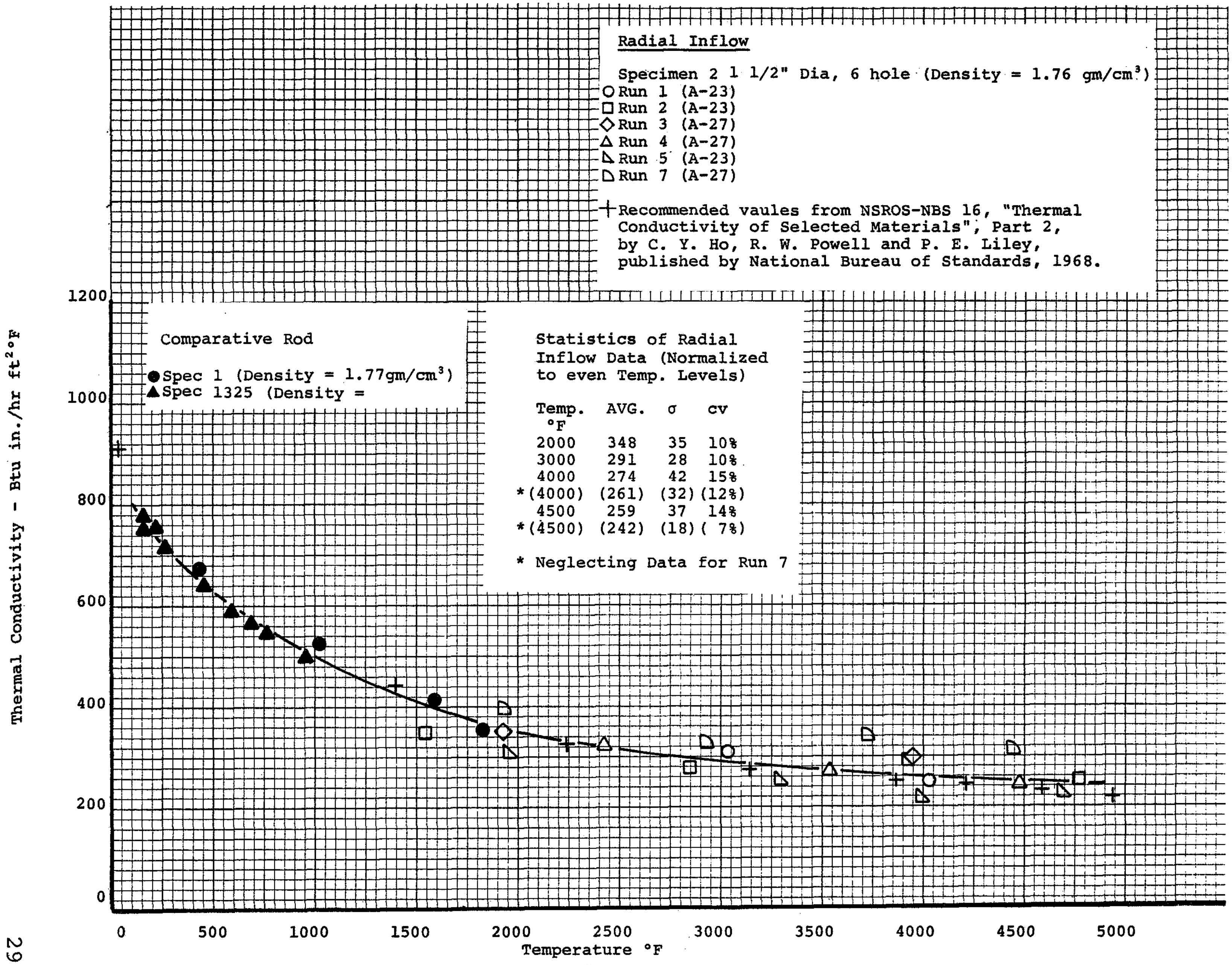

Figure A7. Thermal conductivity of ATJ graphite, with grain

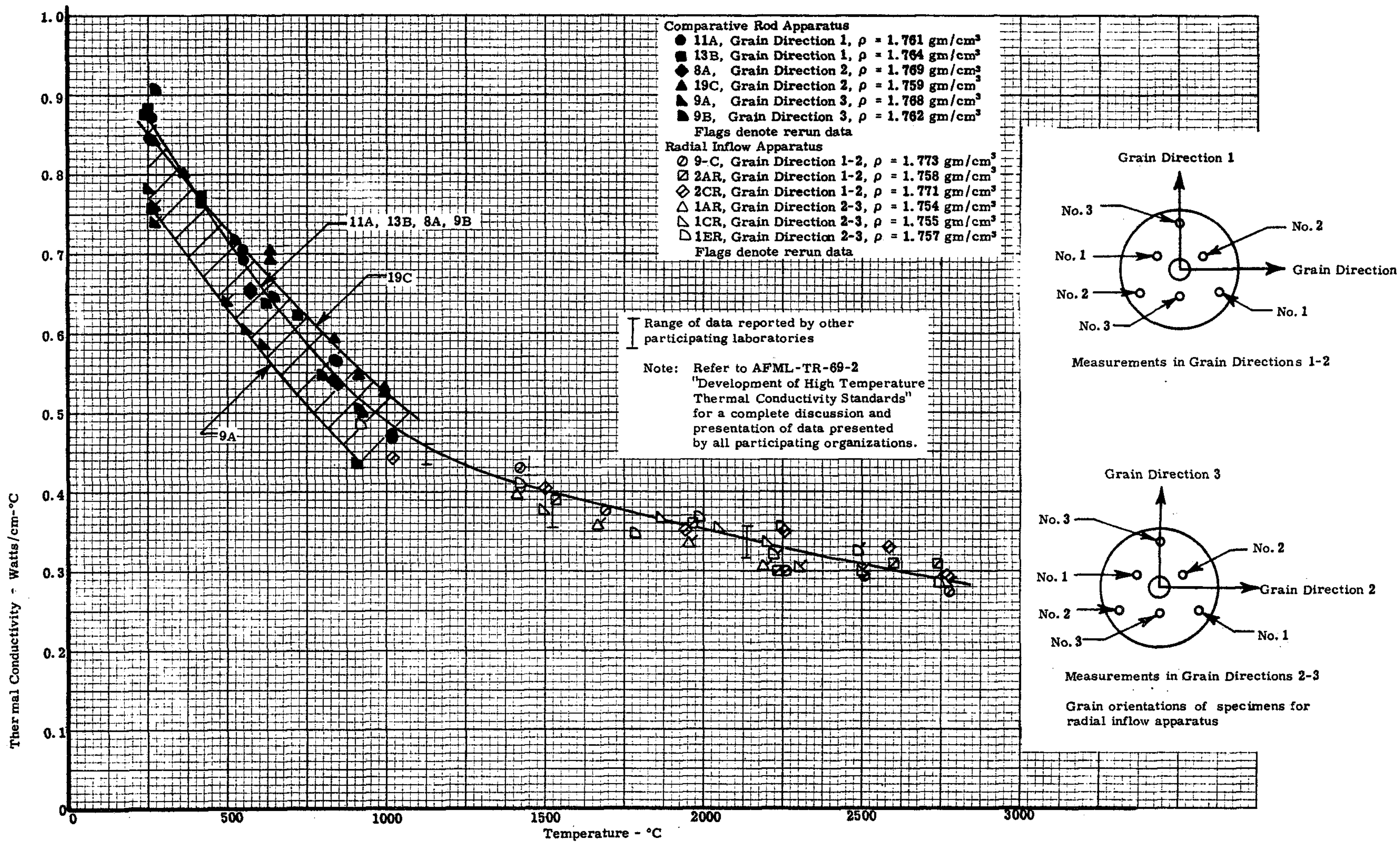

Figure A8. Thermal conductivity of AXM-5Q1 graphite

APPENDIX B

HIGH TEMPERATURE FURNACE WITH ENVIRONMENTAL CAPABILITY

A furnace is available for operation to 4500°F within the environmental pressure range of about 15×10^{-3} torr to 200 psig in inert atmospheres. The 4500°F temperature limit applies to operation under vacuum conditions and can be extended to over 5000°F for pressures above atmospheric. This furnace utilizes an electrically heated helical graphite heating element held between two water cooled copper electrodes. The hot zone of the heater is 2-5/8 inches in diameter by 6 inches long. A liner, radiation shield, also made of graphite, which is slightly larger than the heater tube serves to contain a carbon fiber blanket insulation between the liner and the water cooled shell on the outside. The entire heating element is enclosed within a leak tight shell (see Figure B1) which is designed to provide the range of environmental conditions given above. The several openings in the furnace are sealed with either O-rings or Conax[R] fittings. Ball type valves with Teflon seats are used at all places in the system where an open-close capability is required.

Included in the openings in the furnace are: (1) a sight port with a sapphire window for viewing the center of the specimen, (2) gland type fittings for centering and sealing a water-calorimeter through the furnace (for conductivity measurements), (3) a top plate with several sapphire sight windows for viewing down into the specimen assembly in the axial direction (the top plate can be modified to accomodate sight holes at different locations), (4) thermocouple feedthroughs for making temperature measurements inside the furnace with thermocouples and (5) ball valves for connecting pressure gages to the system.

The heater tube operates at low voltage and high current, about 15 volts at 1500 amps. Power is supplied from a 220 V ac line to an auto transformer which feeds power to water cooled copper electrodes.

The vacuum system for the furnace is a Welch 15 cfm mechanical pump and an NRC HS4 diffusion pump rated at 750 l/sec. Pressures (under vacuum) are monitored by means of McLeod and thermocouple gages. The thermocouple gages serve as system monitors.

Pressurization is achieved by regulation of a bottled gas supply. Pressures are measured with a laboratory-type bourdon pressure gage which is accurate to within about ±2 psi.

The furnace has primarily been used for thermal conductivity measurements but is quite versative for other measurements. For example, additional sight ports are available for measuring thermal expansion optically under vacuum conditions. These ports are aligned so that the ends of a specimen can be illuminated from one side and viewed from the other. Also, the furnace has been used for such tasks as vacuum impregnation with molted metals.

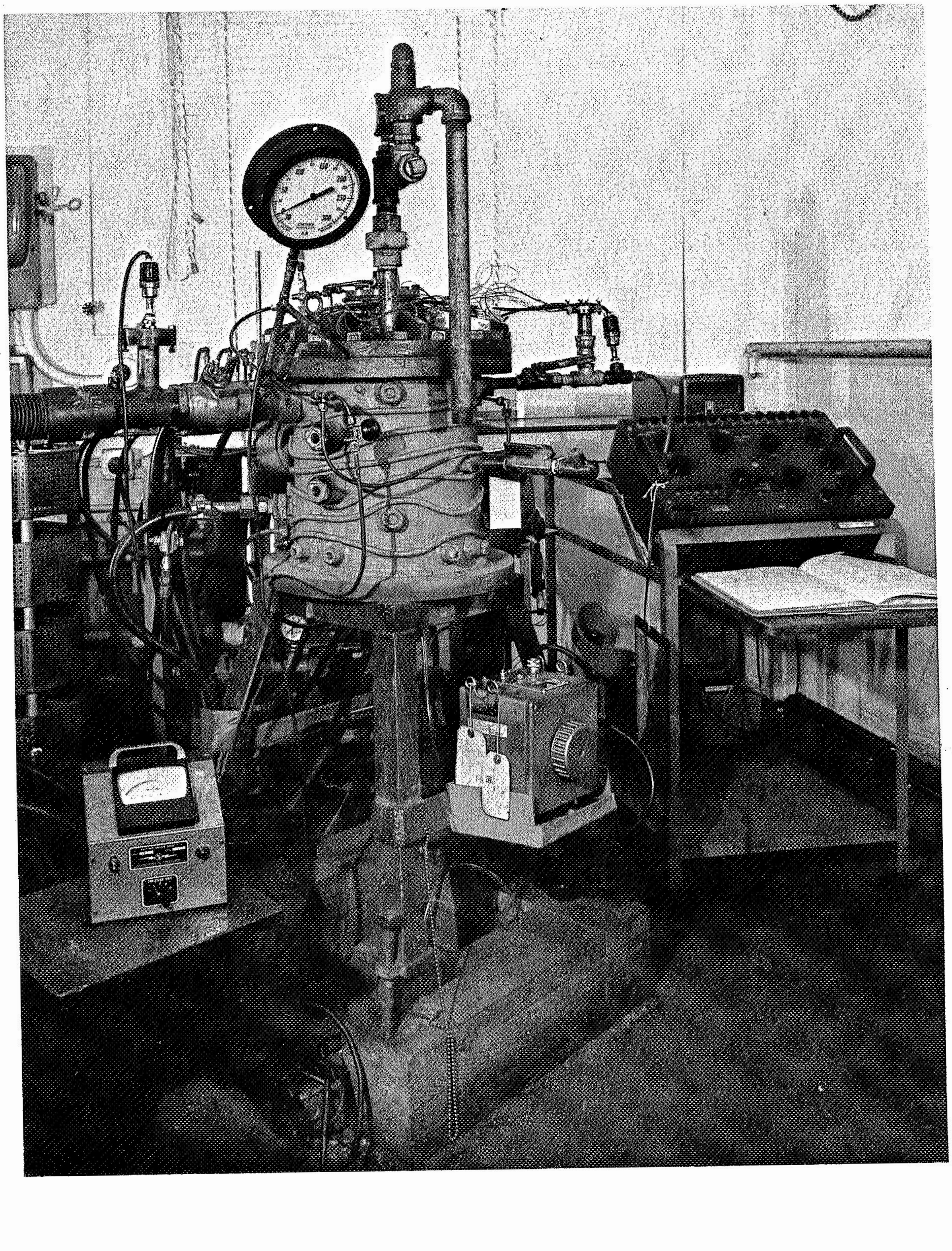

Figure B1. Picture of environmental furnace (capable of operation from 15 microns to 200 psig)

APPENDIX C

HEAT CAPACITY TO 5500°F

The apparatus used for heat capacity employs the drop technique in which the specimen is heated in a furnace and then dropped into an ice calorimeter. The calorimeter contains a cup surrounded by a frozen ice mantle. Water at an inlet temperature of 32°F is circulated through an annulus surrounding the mantle in order to absorb heat leak from the surroundings. The entire system is insulated with glass wool. The enthalpy of the specimen is sensed as a change in volume of the water-ice system of the calorimeter as the ice melts. The annulus containing the flooded ice mantle communicates with the atmosphere through a mercury column in order that the change in volume can be read directly in a burette. An assembly drawing of the calorimeter is shown in Figure C1 and a picture of the calorimeter is shown in Figure C2. A picture of the ice mantle is shown in Figure C3. The specimen nominally is 3/4" diameter x 3/4" long.

The specimen is placed in either a graphite or stainless steel basket and heated in the furnace in a controlled atmosphere such as helium. The specimen and basket are dropped into the calorimeter and the volume change due to the resultant melting of ice is measured. The flutter valve immediately above the cup and the diaphragm valve immediately below the furnace are major features of the apparatus since the first blocks off radiation losses from the specimen up the drop tube, and the second blocks radiation gains from the furnace down the drop tube just prior to dropping. The volume changes due to the baskets are measured and correction curves are established. Separate basket calibration minimizes the radiation error accompanying drop techniques. These errors are reported to be only about 0.5%[1]. Our theoretical calculations indicate even smaller errors from this source.

The heat necessary to melt enough ice to cause a volume change of 1cc has been established by the U. S. National Bureau of Standards[2] at 3.487 Btu. This value is reported as the theoretical one for any ice calorimeter. Figure C4 shows a typical curve of mercury displacement

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[1] Furukawa, G. T., Douglas, McCoskey, and Ginnings, "Thermal Properties of Aluminum Oxide from 0° to 1200°K."

[2] Ginnings, D. C. and R. J. Corruccini, "An Improved Ice Calorimeter," NBS Research Journal, Vol. 38 1947, p. 583.

versus time for one drop using a synthetic sapphire specimen. The correction for the stainless steel basket is subtracted from the measured mercury displacement and the result used to calculate specimen enthalpy above 32°F. The heat capacity, which is by definition the slope of the enthalpy versus temperature curve, is determined both graphically and analytically. The analytical approach is to fit the enthalpy data to an equation of the form

$$h_{32} = aT + bT^2 + cT^{-1} + d \qquad (1)$$

using a least squares technique. The derivative of this equation, the heat capacity, is computed with the constant "c" adjusted so that the analytical solution agrees with the graphical solution at 5000°F.[3] This technique is similar to that of Kelley in forcing the heat capacity equation through a known value.[4] The equations are developed using a digital computer.

A compilation of all errors has indicated that the apparatus is accurate to well within 5% uncertainty over the entire temperature range. Comparison of Southern Research Institute data on copper, Linde synthetic sapphire, and ATJ graphite all have confirmed this value.

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[3] Pears, C. D., and J. G. Allen, "The Thermal Properties of Twenty-six Solid Materials to 5000°F or Their Destruction Temperatures," ASD-TDR-62-765.

[4] Kelley, K. K., "Contributions to Data on Theoretical Metallurgy," Vol. XIII, High Temperature Heat Content, Heat Capacity, and Enthalpy Data for the Elements and Inorganic Compounds, USBM 584, Nov. 1958

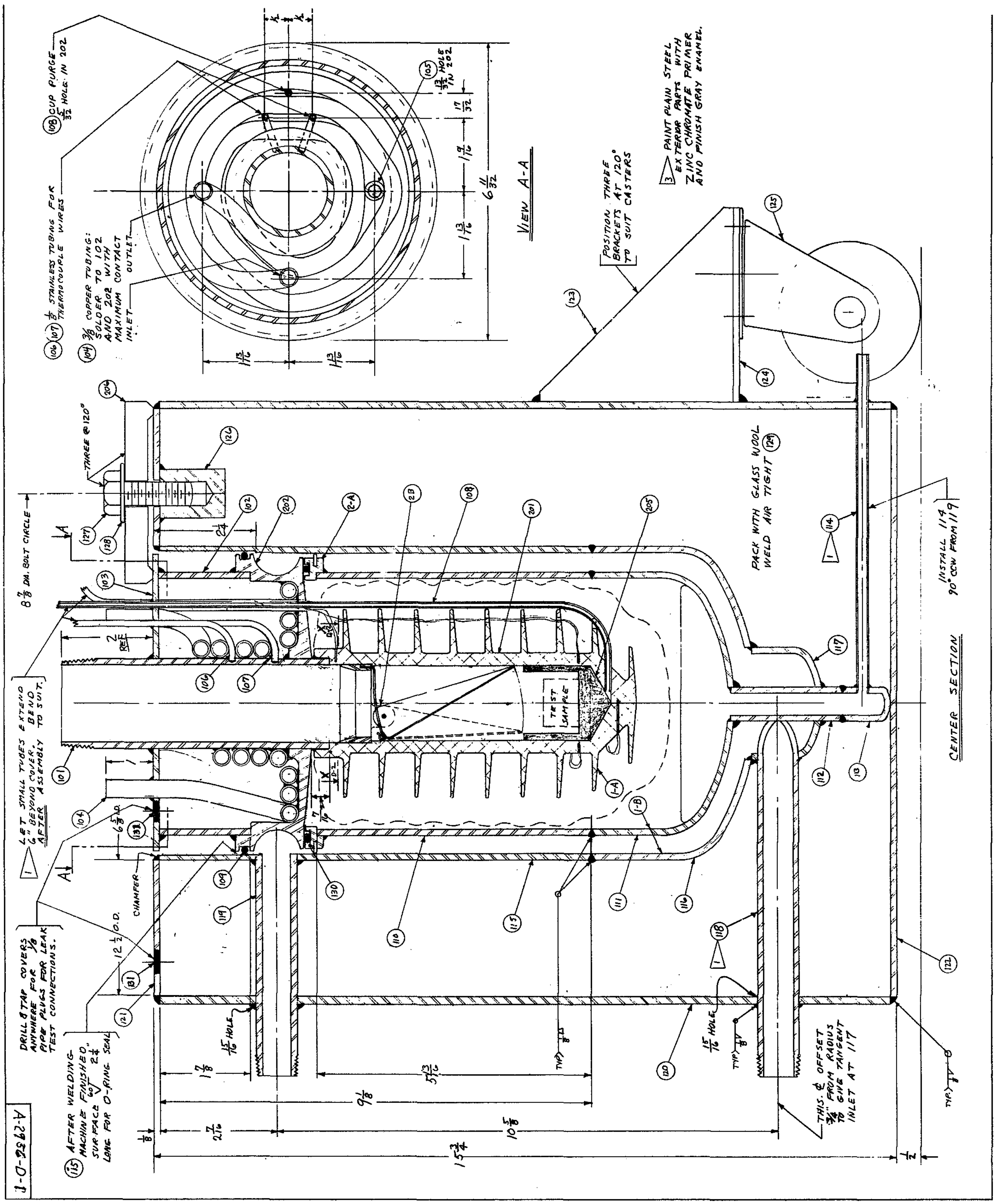


Figure C1. Ice calorimeter, drop type - specimens to 5500°F

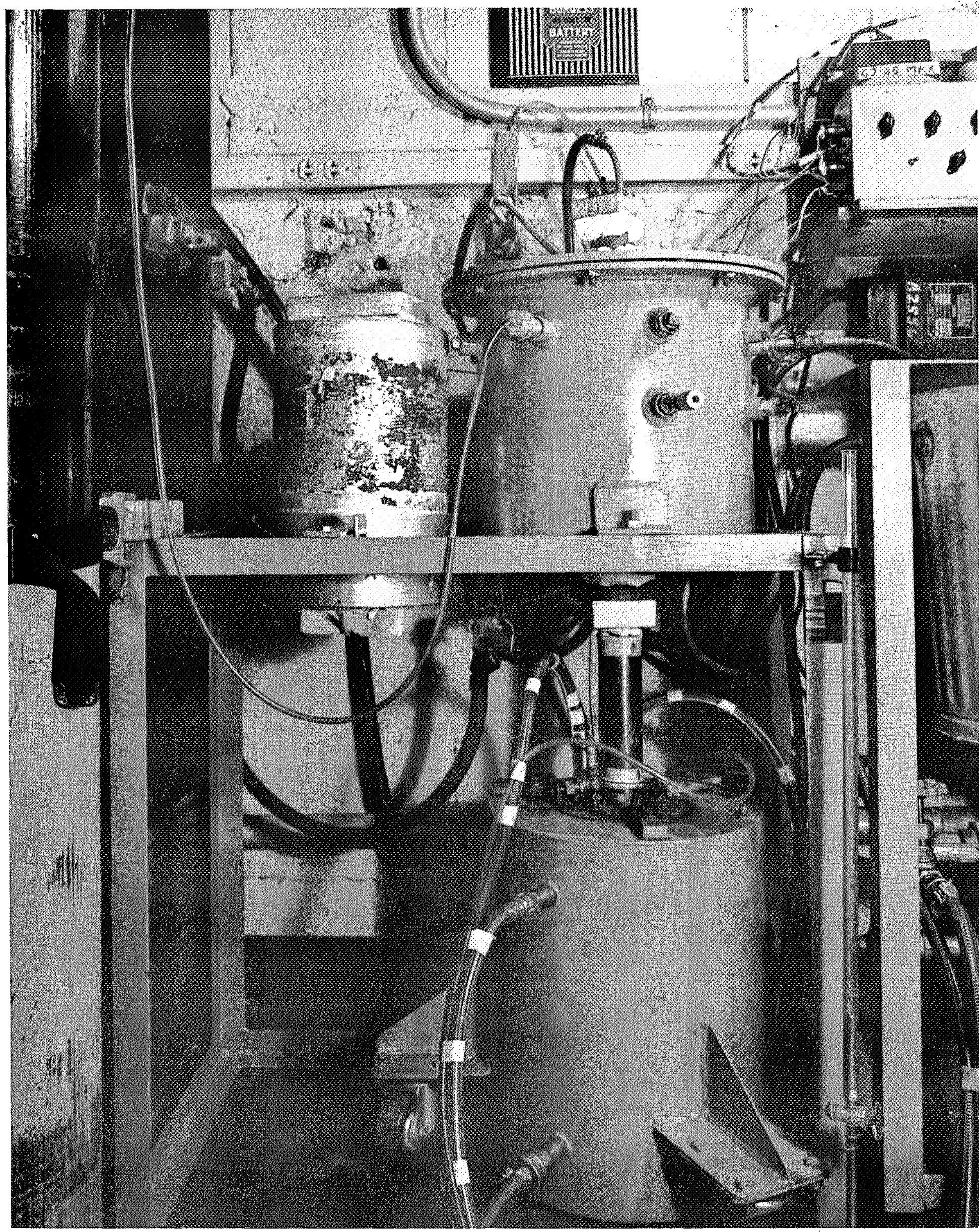


Figure C2. Picture of heat capacity equipment with drop shield tube in place

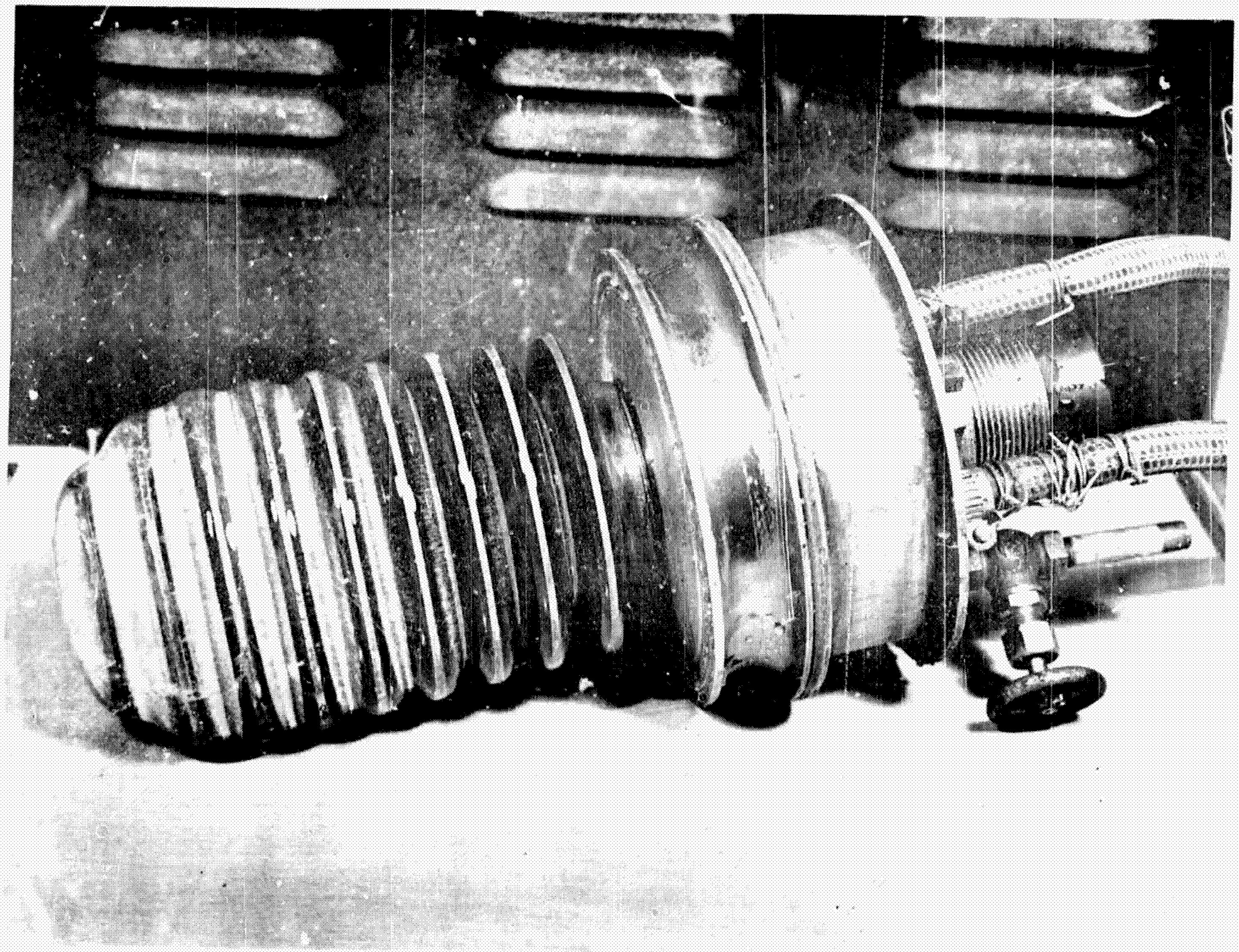


Figure C3. Picture of ice mantle in heat capacity ice calorimeter

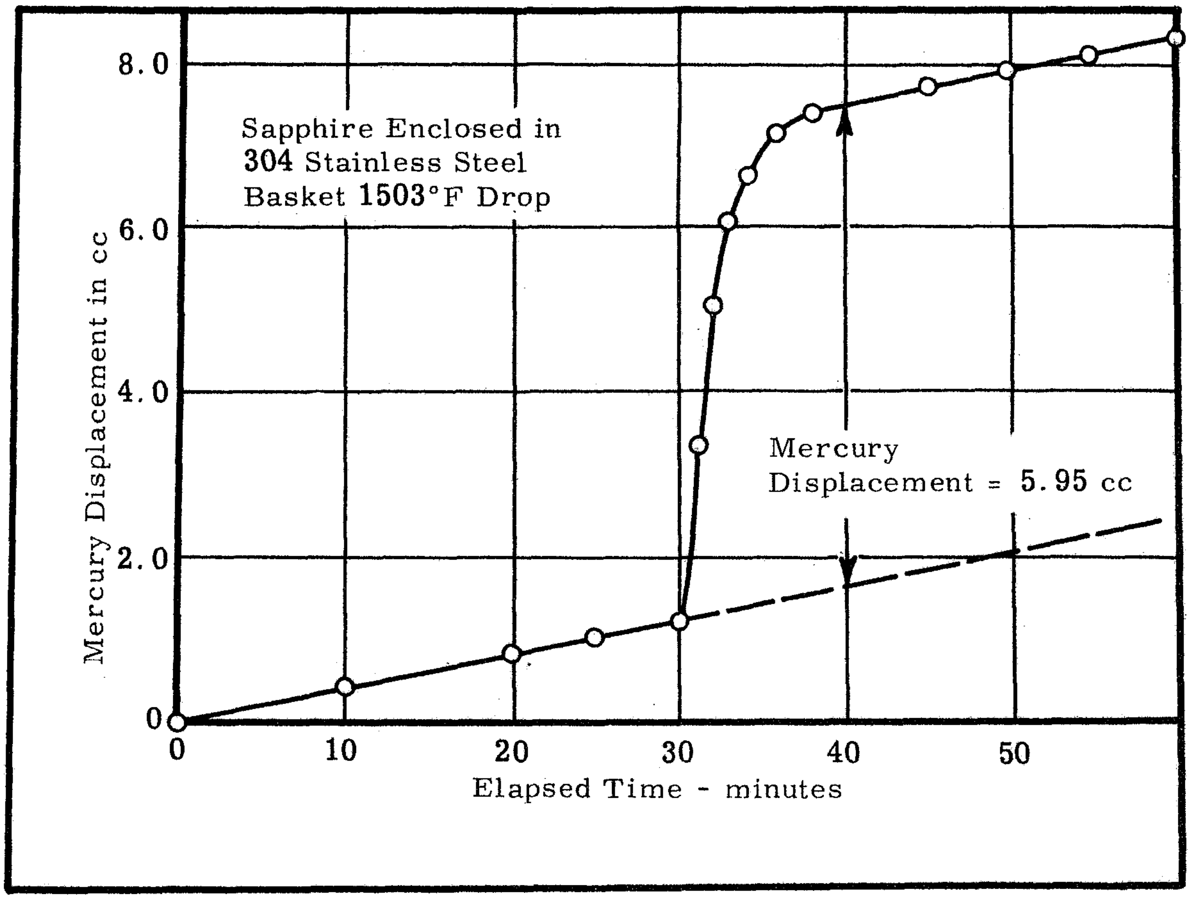


Figure C4. Mercury displacement due to sapphire and 304 stainless steel basket

APPENDIX D

THERMAL EXPANSION TO 5500°F

Thermal expansion is measured in a graphite tube dilatometer developed by Southern Research Institute for performance to 5500°F, see Figure D1. The specimen required is about 1/2" diameter and 3" long, although the exact size can vary somewhat if it appears desirable from the standpoint of specimen availability. Specimens 3/4" in diameter and only 1/4" thick can be evaluated, but with a reduced precision. Discs can be stacked to provide more length in many cases. Of course, specimens can always be pinned together from smaller pieces to provide both length and columnar strength.

In the dilatometer, the specimen rests on the bottom of the cylinder with a graphite extension rod resting on the specimen to extend to the top of the cylinder. When required, tungsten pads are inserted at the ends of the specimen to eliminate graphite diffusion from the dilatometer parts into the specimen. This entire assembly is inserted into one of the 5000°F furnaces described in another brochure.

The motion of the specimen is measured by a dial gage attached to the upper end of the cylinder with the stylus bearing on the extension rod. The system accurately indicates total motions of 0.0001" - or less than 0.00004" per inch of specimen.

Either a helium or an argon environment can be employed. Nitrogen has been used on occasion. The equipment will permit operation at hard vacuums, but this procedure is rarely used.

A CS graphite, which has a fairly low expansion relative to other grades of graphite, is used as the material for the dilatometer. Prior to calibrations, the dilatometers are heat soaked to a temperature several hundred degrees above the maximum temperature to which they would be exposed during normal service. Dimensional stability is confirmed by measuring the lengths of the dilatometer tube and rod after each run. Past experience has shown that following the initial heat soak the expansion is reproducible in subsequent repeated cycles to lower temperatures. Reproducibility is also confirmed by repeated runs on standards.

To calibrate the dilatometers we have developed in-house primary and secondary standards of ATJ graphite. ATJ graphite was selected as a standard because of our vast experience with it, its stability after repeated exposure to high temperatures, and its relatively low expansion.

The true expansion of the primary standard was determined by a direct optical technique using a traveling Gaertner telescope. The total error in the telescope readings, based on calibration data, was estimated to be 0.2 x 10^{-3} in./in. For the direct optical measurements, the 3.5 in. long specimen was heated in a graphite furnace, and the expansion was determined by sighting on "knife" edges machined on the ends of the specimen. Typically a total of 11 runs have been made in two different furnaces both in vacuum and helium environments. The two environments are used to check effects of refraction as reported in the literature. The same standard was then machined to the configuration of a regular dilatometer specimen and several runs were made in our precision quartz dilatometers. The optical expansion data were fitted to a quadratic equation over the temperature range from 2500°F to 5000°F using the method of least squares and statistically analyzed to determine the uncertainty (primarily the scatter). Below 2500°F, the quartz dilatometer data were fitted by hand since the uncertainty of this apparatus has been well established, and the imprecision is small (<0.1 x 10^{-3} in./in.). A typical plot of all data points with the curve fit is shown in Figure D2.

A check of the expansion of the standard was obtained by making runs on round robin specimens of various graphites and synthetic sapphire which had been previously evaluated by others, including the National Bureau of Standards. Our data on these specimens agreed within a 2.5 percent random difference with the data reported by the other laboratories.

After establishing the expansion of the ATJ standard, several graphite dilatometers were then calibrated by making runs on this standard. These dilatometers were used to establish the expansion of secondary standards (also ATJ graphite) which are used to calibrate new dilatometers and to make periodic checks on dilatometers currently in service. This use of secondary standards thus minimizes the wear and tear on the primary standard and prolongs its life.

Table D1 lists the uncertainties in the dilatometer measurements in 10^{-3} in./in. Observe that most of the uncertainty is in the expansion of the standard and includes both random and systematic uncertainties. Other sources of uncertainty, resulting from such factors as dial gage and temperature measurement, are small amounting to less than 0.2 x 10^{-3} in./in. at any temperature. The precision in the dilatometer measurements is quite good and amounts to about 0.1 x 10^{-3} in./in. From Table D1, it can be seen that the maximum total uncertainty, which occurs at a temperature of 4500°F, is ±0.45 x 10^{-3} in./in. For a low expansion graphite, such as ATJ, this amounts to an uncertainty of ±4.5 percent at 4500°F (see Figure D2). For graphites having higher expansions, the percentage uncertainty would be lower.

TABLE D1

Uncertainty in Thermal Expansion Measurements Made in Graphite Dilatometers

Temperature °F	Uncertainty in Expansion of Standard in 10^{-3} in./in. — Random Uncertainty (See Note 1)	Uncertainty in Expansion of Standard in 10^{-3} in./in. — Systematic Uncertainty (See Note 2)	Random Uncertainty in Dilatometer Measurements in 10^{-3} in./in.	Total Uncertainty in Dilatometer Measurements from all Sources in 10^{-3} in./in. +	Total Uncertainty in Dilatometer Measurements from all Sources in 10^{-3} in./in. −
500	±0.04	0	±0.03	0.05	0.05
1000	±0.04	0	±0.03	0.05	0.05
1500	±0.05	0	±0.04	0.07	0.07
2000	±0.17	+0.03	±0.05	0.21	0.18
2500	±0.11	+0.20	±0.07	0.33	0.13
3000	±0.12	+0.21	±0.08	0.35	0.14
3500	±0.19	-0.02	±0.10	0.21	0.23
4000	±0.14	-0.09	±0.12	0.18	0.27
4500	±0.14	±0.25	±0.14	0.45	0.45
5000	±0.19	-0.12	±0.16	0.25	0.37

Notes: 1. 95% confidence limits.
2. Represents deviation between average measured value and least squares curve through all data.

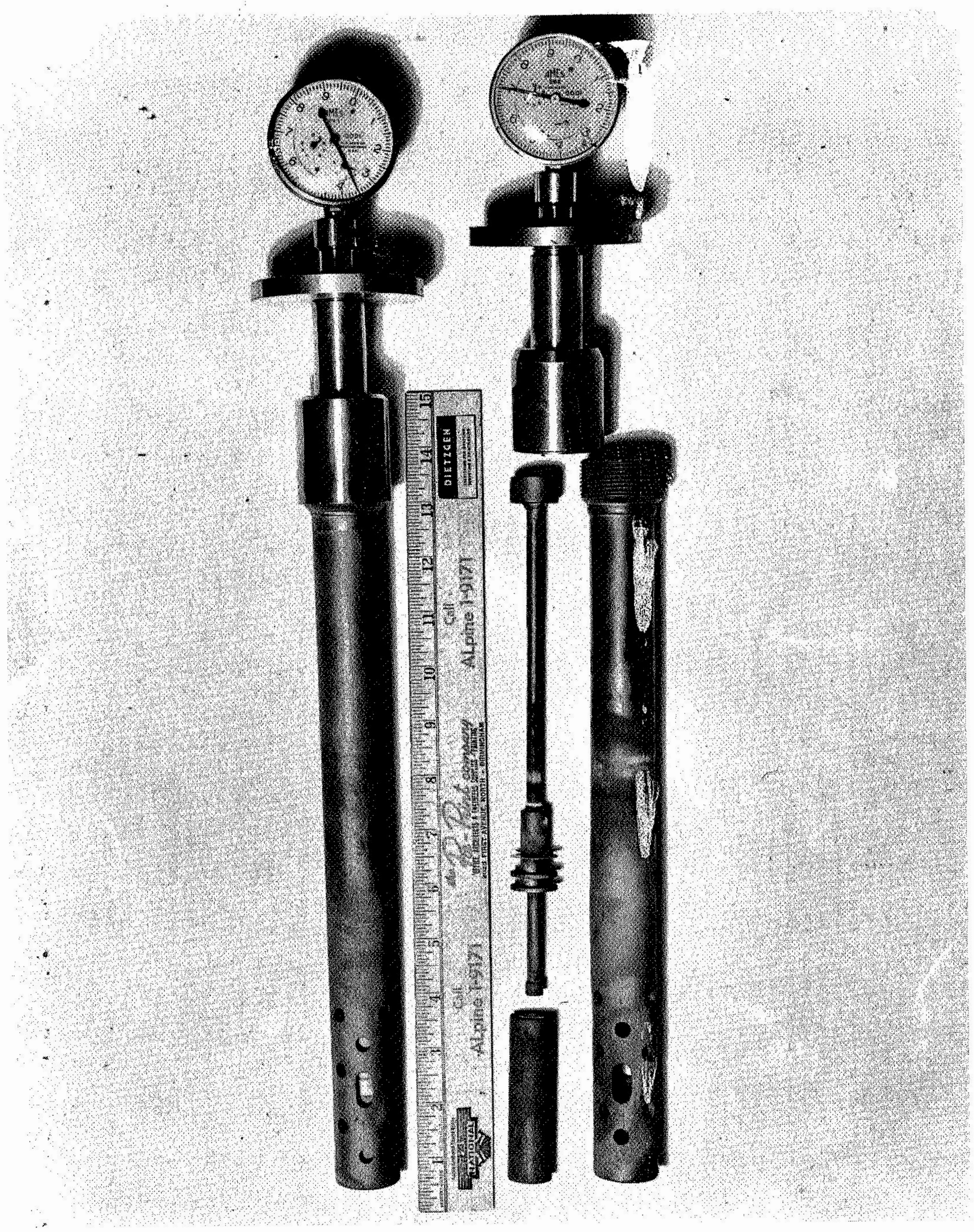


Figure D1. Picture of the graphite dilatometer tubes for measuring thermal expansion to 5500°F

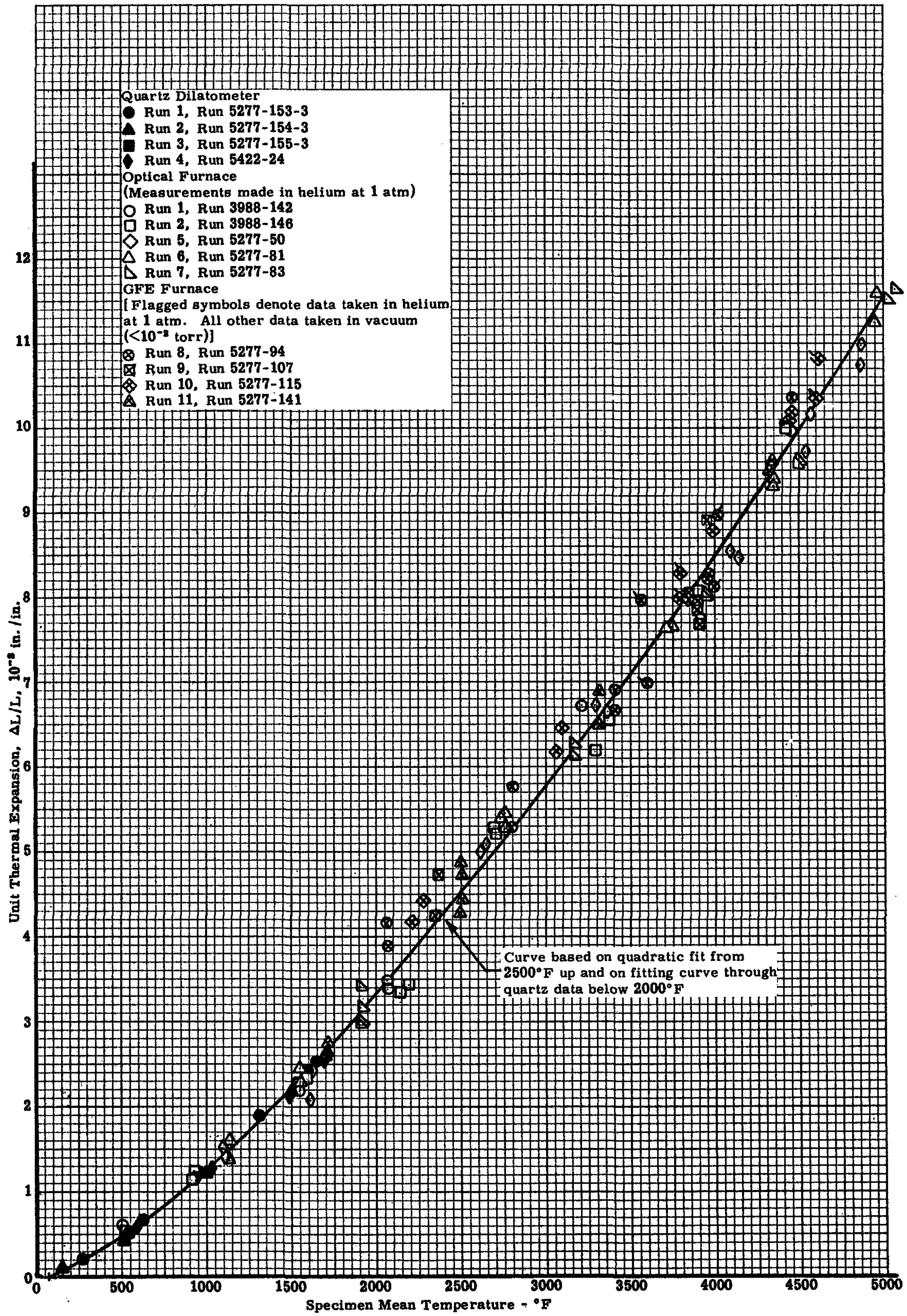


Figure D2. Thermal expansion of ATJ graphite (wg) Standard No. 5 by in-house optical calibration

TABLE 1

INFORMATION ON THREE RIGIDIZED INSULATORS

Material	Density gm/cm^3	Firing temperature K	Firing time hr
Alumina-Silica-Chromia [1]	0.216 to 0.264 range for 10 pieces	1533	1
Mullite [1]	0.228 to 0.255 range for 10 pieces	1589	1
LI-1500 [2]	0.277 gross on 1 block	----	----

1. Manufactured by McDonnell-Douglas Astronautics Company - East
2. Manufactured by Lockheed Missiles and Space Company

TABLE 2

SUMMARY OF DENSITY MEASUREMENTS ON ALUMINA-SILICA-CHROMIA AND LI-1500 RIGIDIZED INSULATIONS

Material and Blank No.	Initial blank measurements						Remarks
	Length cm	Width cm	Thickness cm	Weight g	Volume cm^3	Density gm/cm^3	
Alumina-Silica-Chromia (Green)							
No. 1A	10.178	10.178	3.147	72.2022	325.994	0.2214	7" ASTM-1
No. 1B	10.165	10.180	3.150	73.7486	325.931	0.2262	EXP - Enthalpy
No. 1C	10.079	10.160	3.150	69.7378	322.517	0.2162	7" ASTM-1
No. 1D	10.160	10.160	3.150	70.1431	325.118	0.2157	
No. 2B	10.140	10.165	3.109	70.3388	320.441	0.2195	EXP-Enthalpy
No. 2C	10.097	10.160	3.109	70.1402	318.917	0.2199	
No. 2D	10.147	10.165	3.129	72.6357	322.778	0.2250	
No. 3B	10.160	10.160	3.157	81.0967	325.905	0.2488	Surface Different
No. 3C	10.165	10.193	3.170	85.3320	328.443	0.2598	7" ASTM-2
No. 3D	10.168	10.196	3.172	86.8236	328.870	0.2640	7" ASTM-2
LI-1500 White with Green Surface on One Side (Call Green Side Top of Block)							
	15.222	15.230	6.802	437.0	1576.94	0.2771	

TABLE 3

SUMMARY OF DENSITY MEASUREMENTS ON SLABS OF MULLITE FIBER RIGIDIZED INSULATION

Material and Blank No.	Initial blank measurements						Remarks
	Length cm	Width cm	Thickness cm	Weight g	Volume cm^3	Density gm/cm^3	
Mullite Fiber Rigidized (White)							
No. 1A	10.112	10.109	3.152	72.7905	322.215	0.2259	3" ASTM-1
No. 1C	10.122	10.112	3.155	73.8888	322.88	0.2288	EXP-enthalpy
No. 1D	10.122	10.114	3.160	73.7960	323.481	0.2281	
No. 2A	10.122	10.112	3.142	81.8050	321.580	0.2544	EXP-enthalpy
No. 2B	10.112	10.112	3.160	80.5930	323.075	0.2494	
No. 2C	10.117	10.117	3.157	82.3447	323.140	0.2548	
No. 2D	10.102	10.122	3.152	80.0812	322.296	0.2484	
No. 3B	10.122	10.122	3.162	76.2205	323.985	0.2353	7" ASTM-2
No. 3C	10.122	10.124	3.167	77.7213	323.985	0.2398	7" ASTM-2
No. 3D	10.119	10.124	3.167	77.3160	324.506	0.2382	

TABLE 4

THERMAL CONDUCTIVITY OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN AIR (ASTM C177 GUARDED HOT PLATE, 7 INCHES DIAMETER)

Average specimen mean temperature K	Total heat input watts	Average specimen ΔT K	Specimen thickness[2] cm	Specimen thermal conductivity W/m-K	Time to temperature[3] hr
Specimen AL-ASTM-1A-1C-1 Run 5880-9-7					
Bulk density: Disc 1C1 Top 0.215 gm/cm^3; Initial weight: 33.3418 g; Final weight: 32.9913 g					
Disc 1A1 Bottom 0.226 gm/cm^3; Initial weight: 35.4068 g; Final weight: 34.9021 g					
Gum Rubber Filler					
134	6.06	68.48	0.612	0.0335	5.00
125	6.22	70.71	0.612	0.0332	7.00
127	6.22	65.17	0.612	0.0360	7.50
125	6.22	69.91	0.612	0.0336	8.00
191	19.76	158.36	0.612	0.0470	3.00
192	19.76	157.79	0.612	0.0473	3.50
249	7.62	49.68	0.612	0.0578	7.00
250	7.62	49.57	0.612	0.0580	7.50
Fiberfrax Filler					
332	5.94	31.17	0.605	0.0711	4.25
333	5.94	31.72	0.605	0.0698	4.50
385	13.30	68.66	0.605	0.0722	6.00
386	13.30	68.97	0.605	0.0720	6.5
459	14.68	74.97	0.605	0.0730	4.25
458	14.70	74.45	0.605	0.0737	5.00
513	20.57	93.94	0.605	0.0816	2.25
513	20.57	94.07	0.605	0.0815	2.50

Notes:

1. Diameter of central heater = 10.16 cm (Area - 81.07 cm^2)
2. Thermal conductivity values based on measured thickness at each temperature level.
3. Time to temperature implies the time elapsed between adjustment of power and obtaining data.

TABLE 5

THERMAL CONDUCTIVITY OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN AIR (ASTM C177 GUARDED HOT PLATE, 7 INCHES DIAMETER)

Average specimen mean temperature K	Total heat input watts	Average specimen ΔT K	Specimen thickness[2] cm	Specimen thermal conductivity W/m-K	Time to temperature[3] hr
Specimen AL-ASTM-3C-2 & 3D-2 Run 5880-41-7					
Bulk density: Disc 3C2 Top 0.263 gm/cm³; Initial weight: 41.2473 g; Final weight: 40.7558 g					
Disc 3D2 Bottom 0.270 gm/cm³; Initial weight: 42.2229 g; Final weight: 41.7347 g					
Gum Rubber Filler					
159	12.93	111.73	0.627	0.0448	1.50
158	13.06	109.90	0.627	0.0460	2.00
158	13.21	109.65	0.627	0.0466	2.75
215	27.42	180.74	0.627	0.0587	3.75
216	27.42	179.75	0.627	0.0590	4.50
280	15.43	86.51	0.627	0.0691	3.00
280	15.25	87.17	0.627	0.0678	3.75
Fiberfrax Filler					
365	9.42	49.77	0.622	0.0725	4.00
363	9.42	48.34	0.622	0.0748	5.50
428	19.37	91.14	0.622	0.0816	4.25
428	19.37	90.96	0.622	0.0818	5.00
462	26.59	114.00	0.622	0.0895	1.75
463	26.49	113.43	0.622	0.0897	4.25
534	24.56	95.55	0.617	0.0979	5.25
534	24.56	95.70	0.617	0.0978	5.25
598	32.64	119.92	0.617	0.1037	3.50
595	32.65	120.10	0.617	0.1035	4.25

Notes:

1. Diameter of central heater - 10.16 cm (Area - 81.07 cm^2)
2. Thermal conductivity values based on measured thickness at each temperature level.
3. Time to temperature implies the time elapsed between adjustment of power and obtaining data.

TABLE 6

THERMAL CONDUCTIVITY OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN NITROGEN (RADIAL INFLOW APPARATUS - POSITIVE LOAD FURNACE)

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
Spec AL-1 Load Furnace Run 5880-61-A5 Average initial thickness: 0.254 cm Average final thickness: 0.203 cm	9:30	688	F-313	10.46				~1 ATM nitrogen purge with a constant 10 psi compaction loading.
		689	B-316	10.96				
		680	L-311	9.23				
		686	R-314	10.70				
				10.46				
				9.00				
	Average	686	314	10.23	528	0.130	0.256	
	11:30	1111	F-586	24.3				
		1106	B-584	24.9				
		1101	L-589	23.7				
		1106	R-585	25.8				
				25.5				
				24.0				
	Average	1106	586	24.8	810	0.168	0.256	
	1:25	1476	F-710	44.8				
		1463	B-702	43.4				
		1464	L-724	45.1				
		1459	R-699	43.4				
				43.4				
	Average	1466	709	44.0	1105	0.234	0.244	
	3:20	1626	F-690	50.7				
		1618	B-686	52.4				
		1623	L-720	52.2				
		1614	R-689	51.6				
				51.6				
				53.3				
	Average	1620	696	52.2	1264	0.257	0.221	

TABLE 7

THERMAL CONDUCTIVITY OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN VACUUM AND NITROGEN (RADIAL INFLOW APPARATUS - ENVIRONMENTAL FURNACE)

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
Spec AL-2	9:15	813	F-267	6.21				
Run 5867-76		813	B-266	6.15				
Average Density		818	L-270	6.62				
0.219 gm/cm		785	R-239	6.91				
Average initial				7.15				
thickness:	Average	807	261	6.62	676	0.0549	0.248	3.9×10^{-3} torr
0.254 cm								
Average final								
thickness:	10:15	796	F-203	11.05				
0.229 cm		789	B-196	10.02				
		785	L-192	10.26				
		785	R-193	10.67				
				10.49				
	Average	789	196	10.49	690	0.116	0.248	~1 ATM nitrogen purge
	12:00	1156	F-346	14.30				
		1155	B-343	13.48				
		1156	L-341	13.89				
		1153	R-339	13.60				
				14.86				
	Average	1155	342	14.04	983	0.0872	0.244	2.0×10^{-3} torr
	1:15	1128	F-275	25.2				
		1122	B-268	24.6				
		1118	L-264	23.2				
		1118	R-264	24.3				
				23.7				
	Average	1121	268	24.4	985	0.195	0.245	~1 ATM nitrogen purge

TABLE 7 CONCLUDED

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
	3:40	1425	F-300	20.8				
		1425	B-296	20.5				
		1425	L-286	21.4				
		1422	R-289	21.4				
				21.7				
	Average	1424	292	21.2	1276	0.152	0.241	3.8×10^{-3} torr
	3:45	1454	F-268	32.5				
		1454	B-266	33.7				
		1451	L-261	32.5				
		1451	R-266	33.7				
				35.5				
	Average	1453	265	33.6	1318	0.265	0.240	~1 ATM nitrogen purge

Note: Densities of strips comprising specimen
5a = 0.224 gm/cm^3; 7a = 0.214 gm/cm^3
6a = 0.238 gm/cm^3; 8a = 0.200 gm/cm^3

TABLE 8

THERMAL CONDUCTIVITY OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN VACUUM AND NITROGEN (RADIAL INFLOW APPARATUS - ENVIRONMENTAL FURNACE)

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
Spec AL-3 Run 5867-112-A5 Average density 0.222 gm/cm^3 Average initial thickness: 0.254 cm Average final thickness: 0.232 cm	9:15	812	F-284	8.32				
		833	B-306	8.50				
		836	L-307	8.41				
		832	R-304	8.94				
				9.17				
	Average	828	301	8.64	678	0.0617	0.246	1.5×10^{-3} torr
	10:30	786	F-207	12.63				
		803	B-222	12.75				
		801	L-220	13.04				
		799	R-218	13.24				
				12.72				
	Average	797	217	12.86	688	0.128	0.248	~1 ATM nitrogen purge
	12:05	---	F----	13.60				
		1097	B-391	13.95				
		1099	L-389	13.83				
		1096	R-387	13.89				
				14.12				
	Average	1097	389	13.89	901	0.0789	0.245	2.2×10^{-3} torr
	1:25	1081	F-295	18.9				
		1081	B-291	20.1				
		1080	L-286	20.1				
		1077	R-286	20.6				
				18.9				
	Average	1080	290	19.7	934	0.146	0.245	~1 ATM nitrogen purge

TABLE 8 CONCLUDED

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
	2:40	1419	F-334	20.2				
		1418	B-330	19.0				
		1420	L-321	20.2				
		1418	R-319	19.8				
				19.0				
	Average	1419	326	19.7	1254	0.126	0.241	5.8×10^{-3} torr
	3:50	1447	F-288	34.0				
		1447	B-283	34.3				
		1447	L-276	32.8				~1 ATM nitrogen purge
		1445	R-281	33.4				
				33.4				
	Average	1446	282	33.4	1303	0.248	0.240	

Note: Densities of strips comprising specimen
9a = 0.250 gm/cm^3; 11a = 0.206 gm/cm^3
10 a = 0.229 gm/cm^3; 12a = 0.204 gm/cm^3

TABLE 9

THERMAL CONDUCTIVITY OF MULLITE FIBER RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN AIR (ASTM C177 GUARDED HOT PLATE, 3 INCHES DIAMETER)

Average specimen mean temperature K	Total heat input watts	Average specimen ΔT K	Specimen thickness[2] cm	Specimen thermal conductivity W/m-K	Time to temperature[3] hr
Specimen MUL-ASTM-1A	Run 5880-6-3				
Bulk Density: Disc 2	Top 0.227 gm/cm^3; Initial weight: 7.5515 g; Final weight: 7.1409 g				
Disc 1	Bottom 0.219 gm/cm^3; Initial weight: 7.2776 g; Final weight: 6.6730 g				
Gum Rubber Filler					
152	2.61	105.18	0.592	0.0401	1.25
152	2.61	106.58	0.592	0.0397	1.50
153	2.61	107.38	0.592	0.0394	2.00
211	5.25	180.94	0.592	0.0470	3.00
211	5.25	180.50	0.592	0.0470	3.50
266	2.58	78.10	0.592	0.0535	3.50
266	2.59	78.52	0.592	0.0534	4.25
Fiberfrax Filler					
394	2.91	80.34	0.577	0.0571	5.75
393	2.91	80.49	0.577	0.0570	6.00
463	5.95	130.33	0.577	0.0720	5.50
464	5.95	131.20	0.577	0.0715	5.75
559	5.02	108.59	0.577	0.0728	6.00
559	5.03	108.66	0.577	0.0730	6.25

Notes:

1. Diameter of central heater = 4.826 cm (Area = 18.29 cm^2)
2. Thermal conductivity values based on measured thickness at each temperature level.
3. Time to temperature implies the time elapsed between adjustment of power and obtaining data.

TABLE 10

THERMAL CONDUCTIVITY OF MULLITE FIBER RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN AIR (ASTM C177 GUARDED HOT PLATE, 7 INCHES DIAMETER)

Average specimen mean temperature K	Total heat input watts	Average specimen ΔT K	Specimen thickness[2] cm	Specimen thermal conductivity W/m-K	Time to temperature[3] hr
Specimen MUL-ASTM-3B & 3C Run 5880-21-7					
Bulk density: Disc 3C Top 0.238 gm/cm^3; Initial weight: 37.4010 g; Final weight: 36.9262 g					
Disc 3B Bottom 0.234 gm/cm^3; Initial weight: 36.7339 g; Final weight: 36.2912 g					
Gum Rubber Filler					
148	10.64	102.10	0.632	0.0408	3.50
148	10.64	102.70	0.632	0.0404	4.25
185	18.80	150.30	0.632	0.0487	3.50
185	18.80	150.23	0.632	0.0487	4.50
240	4.98	35.49	0.632	0.0548	3.50
239	4.98	34.64	0.632	0.0561	2.00
282	14.93	87.46	0.632	0.0666	3.00
283	14.87	89.02	0.632	0.0652	4.25
Fiberfrax Filler					
320	3.67	19.92	0.630	0.0715	4.75
320	3.67	20.26	0.630	0.0704	5.25
368	9.63	55.85	0.630	0.0671	5.00
370	9.63	55.54	0.630	0.0673	5.50
430	19.44	94.26	0.630	0.0802	6.50
431	19.69	95.48	0.630	0.0802	7.50
505	21.55	89.30	0.627	0.0934	2.75
508	21.10	89.67	0.627	0.0911	3.50
509	21.17	89.73	0.627	0.0913	4.25
599	29.19	115.85	0.627	0.0975	3.50
597	30.27	115.90	0.627	0.1011	4.50
598	30.27	116.20	0.627	0.1008	5.00

Notes:

1. Diameter of central heater - 10.16 cm (Area - 81.07 cm^2)
2. Thermal conductivity values based on measured thickness at each temperature level.
3. Time to temperature implies the time elapsed between adjustment of power and obtaining data.

TABLE 11

THERMAL CONDUCTIVITY OF MULLITE FIBER RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN VACUUM AND NITROGEN (RADIAL INFLOW APPARATUS - ENVIRONMENTAL FURNACE)

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
Spec MUL-1 Run 5867-60-A29 Average density 0.238 gm/cm^3 Average initial thickness: 0.254 cm Average final thickness: 0.142 cm	8:50	800	F-220	6.39				
		799	B-216	5.30				
		769	L-187	5.63				
		777	R-238	6.39				
				7.21				
	Average	786	215	6.18	679	0.0622	0.248	4.5×10^{-3} torr
	10:00	770	F-179	11.75				
		773	B-178	9.99				
		766	L-173	10.61				
		768	R-176	10.72				
				13.07				
	Average	769	176	11.22	681	0.138	0.248	~1 ATM nitrogen purge
	11:55	1164	F-246	15.5				
		1167	B-246	14.2				
		1164	L-237	15.2				
		1165	R-240	16.4				
				16.5				
	Average	1165	241	15.6	1043	0.137	0.244	5.5×10^{-3} torr
	1:40	1155	F-241	24.3				
		1161	B-237	22.9				
		1151	L-227	22.9				
		1153	R-232	24.3				
				24.0				
	Average	1155	234	23.7	1038	0.216	0.244	~1 ATM nitrogen purge

TABLE 11 CONCLUDED

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
	2:45	1381	F-208	20.8				
		1385	B-198	20.5				
		1381	L-192	20.8				
		1382	R-197	20.5				
				20.5				
	Average	1382	198	20.6	1281	0.219	0.241	7×10^{-3} torr
	3:55	1374	F-234	33.1				
		1381	B-226	33.7				
		1374	L-221	32.5				
		1375	R-225	32.8				
				33.7				
	Average	1376	227	33.2	1260	0.308	0.241	~1 ATM nitrogen purge

Notes:

1. Specimen was exposed to 1810K (Hot Hole) trying to obtain higher temperature data
2. Densities of strips comprising specimen
   1a = 0.268 gm/cm^3; 3a = 0.228 gm/cm^3
   2a = 0.234 gm/cm^3; 4a = 0.223 gm/cm^3

TABLE 12

THERMAL CONDUCTIVITY OF MULLITE FIBER RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN VACUUM AND NITROGEN (RADIAL INFLOW APPARATUS - ENVIRONMENTAL FURNACE)

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
Spec MUL-2 Run 5867-100-A5 Average density 0.240 gm/cm^3 Average initial thickness: 0.254 cm Average final thickness: 0.243 cm	8:50	824	F-207	3.25				
		816	B-201	3.14				
		821	L-205	3.57				
		816	R-199	3.25				
				2.52				
	Average	820	203	3.14	718	0.0333	0.248	1.2 x 10^{-3} torr
	10:30	792	F-172	6.91				
		790	B-169	7.03				
		793	L-171	6.89				
		792	R-171	6.39				
				6.89				
	Average	792	171	6.80	706	0.0859	0.248	~1 ATM nitrogen purge
	12:20	1136	F-238	11.69				
		1135	B-248	12.34				
		1136	L-233	13.27				
		1131	R-227	13.95				
				12.92				
	Average	1135	237	12.83	1015	0.116	0.245	2.5 x 10^{-3} torr
	1:25	1153	F-229	15.1				
		1151	B-222	15.7				
		1149	L-220	14.3				
		1150	R-222	16.1				
				16.2				
	Average	1151	223	15.5	1038	0.147	0.244	~1 ATM nitrogen purge

TABLE 12 CONCLUDED

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
	3:10	1408	F-209	15.8				
		1408	B-223	15.8				
		1410	L-201	15.8				
		1405	R-196	15.4				
				15.5				
	Average	1408	207	15.7	1303	0.158	0.241	4.0×10^{-3} torr
	4:15	1455	F-229	27.0				
		1454	B-223	27.0				
		1454	L-216	26.5				~1 ATM nitrogen purge
		1453	R-219	26.0				
				26.5				
	Average	1454	222	26.5	1341	0.250	0.240	

Note: Densities of strips comprising specimen
5a = 0.264 gm/cm^3; 7a = 0.235 gm/cm^3
6a = 0.236 gm/cm^3; 8a = 0.226 gm/cm^3

TABLE 13

THERMAL CONDUCTIVITY OF LI-1500 RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN AIR (ASTM C177 GUARDED HOT PLATE, 3 INCHES DIAMETER)

Average specimen mean temperature K	Total heat input watts	Average specimen ΔT K	Specimen thickness[2] cm	Specimen thermal conductivity W/m-K	Time to temperature[3] hr
Specimen LI-1500-ASTM Disc 1 & 2 Run 5880-21-3					
Bulk density: Disc 1 Top 0.272 gm/cm^3; Initial weight: 9.3550 g; Final weight: 9.1579 g					
Disc 2 Bottom 0.265 gm/cm^3; Initial weight: 8.8991 g; Final weight: 8.7422 g					
Gum Rubber Filler					
163	2.67	129.38	0.632	0.0356	3.00
164	2.67	130.10	0.632	0.0355	3.50
201	4.13	180.18	0.632	0.0397	5.00
200	4.13	180.28	0.632	0.0397	3.00
266	2.58	78.72	0.632	0.0567	3.25
268	2.58	79.23	0.632	0.0562	4.50
268	2.62	81.35	0.632	0.0557	5.50
Fiberfrax Filler					
315	2.75	94.75	0.638	0.0506	1.50
314	2.75	93.16	0.638	0.0515	2.00
380	2.68	79.35	0.638	0.0588	2.50
382	2.68	81.12	0.638	0.0575	3.00
434	4.01	124.59	0.638	0.0561	2.25
434	4.06	124.28	0.638	0.0570	5.50
526	3.83	114.54	0.632	0.0578	2.50
527	3.83	115.11	0.632	0.0575	3.25

Notes:

1. Diameter of central heater = 4.825 cm (Area - 18.29 cm^2)
2. Thermal conductivity values based on measured thickness at each temperature level.
3. Time to temperature implies the time elapsed between adjustment of power and obtaining data.

TABLE 14

THERMAL CONDUCTIVITY OF LI-1500 RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN AIR (ASTM C177 GUARDED HOT PLATE, 3 INCHES DIAMETER)

Average specimen mean temperature K	Total heat input watts	Average specimen ΔT K	Specimen thickness[2] cm	Specimen thermal conductivity W/m-K	Time to temperature[3] hr
Specimen LI-1500 Discs 3 & 4 Run 5800-41-3					
Bulk density: Disc 4 Top 0.272 gm/cm^3; Initial weight: 9.1122 g; Final weight: 9.0517 g					
Disc 3 Bottom 0.270 gm/cm^3; Initial weight: 9.0639 g; Final weight: 9.0059 g					
Gum Rubber Filler					
150	2.26	117.48	0.635	0.0334	2.75
150	2.26	117.65	0.635	0.0333	3.75
204	4.86	205.24	0.635	0.0410	3.75
204	4.83	205.28	0.635	0.0408	4.50
258	2.40	84.52	0.635	0.0492	2.75
258	2.38	84.09	0.635	0.0491	3.75
Fiberfrax Filler					
369	2.03	72.01	0.632	0.0487	3.75
369	2.03	71.68	0.632	0.0490	4.50
367	2.03	72.59	0.632	0.0482	6.50
413	3.88	116.24	0.632	0.0576	5.00
414	3.88	117.12	0.632	0.0572	6.00
417	3.88	120.28	0.632	0.0557	7.25
483	3.46	105.79	0.632	0.0564	3.00
483	3.49	105.60	0.632	0.0570	3.50
526	4.20	124.68	0.632	0.0582	3.00
526	4.20	124.01	0.632	0.0580	2.50
578	5.05	146.97	0.632	0.0593	2.00
578	5.05	146.85	0.632	0.0593	2.75

Notes:

1. Diameter of central heater - 4.825 cm (Area - 18.29 cm^2)
2. Thermal conductivity values based on measured thickness at each temperature level.
3. Time to temperature implies the time elapsed between adjustment of power and obtaining data.

TABLE 15

THERMAL CONDUCTIVITY OF LI-1500 RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN VACUUM AND NITROGEN (RADIAL INFLOW APPARATUS - ENVIRONMENTAL FURNACE)

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
Spec LI-1500-1	9:50	805	F-384	4.81				
Run 5867-44-69-2		809	B-296	4.51				
		825	L-294	4.69				
Average density 0.260 gm/cm^3		805	R-290	4.78				
				4.51				
Average initial thickness: 0.254 cm	Average	811	291	4.66	665	0.0348	0.249	4.3 x 10^{-3} torr
Average final thickness: 0.111 cm	11:30	795	F-236	10.23				
		793	B-234	10.52				
		799	L-238	10.52				
		791	R-231	11.08				
				10.72				
	Average	794	234	10.61	676	0.0979	0.248	~1 ATM nitrogen purge
	1:00	1151	F-452	11.13				
		1130	B-443	11.63				
		1148	L-436	11.95				
		1132	R-440	11.98				
				12.69				
	Average	1140	442	11.87	919	0.0572	0.245	3.8 x 10^{-3} torr
	2:10	1108	F-354	22.5				
		1094	B-338	20.8				
		1106	L-345	21.3				
		1091	R-332	21.7				
				21.4				
	Average	1100	341	21.0	928	0.132	0.245	~1 ATM nitrogen purge

TABLE 15 CONCLUDED

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
	3:35	1355	F-473	16.3				
		1354	B-478	16.8				
		1362	L-497	17.8				
		1350	R-473	16.8				
				17.4				
		1355	480	17.0	1114	0.0745	0.242	5.3 x 10^{-3} torr
	4:35	1375	F-401	33.1				
		1370	B-397	30.5				
		1376	L-397	31.9				
		1357	R-382	33.4				
				32.5				
	Average	1370	394	32.2	1170	0.172	0.241	~1 ATM nitrogen purge

Notes:

1. Specimen was exposed to 2762K (Hot Hole) while obtaining higher temperature data. Data are not shown due to specimen shrinkage
2. Densities of strips comprising specimen
   1a = 0.264 gm/cm^3; 3a = 0.254 gm/cm^3
   2a = 0.262 gm/cm^3; 4a = 0.260 gm/cm^3

TABLE 16

THERMAL CONDUCTIVITY OF LI-1500 RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION IN VACUUM AND NITROGEN (RADIAL INFLOW APPARATUS - ENVIRONMENTAL FURNACE)

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
Spec LI-1500-2 Run 5867-88-A5 Average density 0.260 gm/cm^3 Average initial thickness: 0.254 cm Average final thickness: 0.237 cm	9:00	821	F-290	5.51				
		830	B-298	5.65				
		824	L-290	6.42				
		824	R-291	6.21				
				6.89				
	Average	825	292	6.15	679	0.0456	0.248	2.5×10^{-3} torr
	10:30	801	F-223	10.28				
		802	B-223	10.02				
		803	L-223	9.93				
		801	R-222	9.93				
				10.64				
	Average	802	223	10.17	690	0.0986	0.248	~1 ATM nitrogen purge
	12:10	1129	F-428	10.05				
		1135	B-432	9.82				
		1130	L-426	9.32				
		1130	R-427	9.67				
				10.02				
	Average	1131	428	9.79	916	0.0487	0.245	1.7×10^{-3} torr
	1:25	1123	F-322	21.8				
		1127	B-322	21.8				
		1126	L-321	22.2				
		1126	R-321	22.5				
				22.5				
		1125	322	22.4	963	0.148	0.245	~1 ATM nitrogen purge

TABLE 16 CONCLUDED

Specimen	Time	Average hot hole temperature K	Average ΔT each strip K	Heat flow to calorimeter watts	Average mean temp. of specimen K	Average thermal conductivity of specimen W/m-K	Average specimen thickness at temp cm	Environment and Pressure
	2:40	1437	F-462	17.2				
		1442	B-464	17.4				
		1447	L-464	17.0				
		1447	R-459	17.2				
				17.1				
	Average	1441	462	17.2	1209	0.0777	0.240	4.7×10^{-3} torr
	4:05	1484	F-356	32.2				
		1489	B-356	32.5				
		1494	L-357	31.4				
		1485	R-350	33.1				~1 ATM nitrogen purge
				33.4				
	Average	1488	354	32.5	1308	0.192	0.240	

Note: Densities of strips comprising specimen
5a = 0.266 gm/cm^3; 7a = 0.260 gm/cm^3
6a = 0.255 gm/cm^3; 8a = 0.262 gm/cm^3

TABLE 17

ENTHALPY OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION
(ADIABATIC CALORIMETER)

Specimen	Drop No.	Initial cup temp. K	Final cup temp. K	Change in cup temp. K	Initial temp. of sample K	Initial wt. of sample g	W_s Final Wt. of Sample g	Enthalpy [1] $h = \frac{k}{W_s}(t_2-t_1)$ Joules/kg	Enthalpy Joules/kg above 303 K	Enthalpy Joules/kg above 273 K
ALU-CPA-2B-1	1	297.92	296.55	-1.37	116	6.6246	6.5922	-1.05×10^5	-1.08×10^5	-0.87×10^5
	2	298.05	297.41	-0.64	224	6.5922	6.5492	-0.49×10^5	-5.24×10^4	-0.31×10^5
	3	299.58	300.29	0.71	367	6.5492	6.5097	0.55×10^5	5.26×10^4	0.74×10^5
	4	298.62	300.63	2.01	474	6.5097	6.4874	1.56×10^5	1.55×10^5	1.76×10^5
	5	298.56	302.08	3.52	585	6.4874	6.4693	2.74×10^5	2.73×10^5	2.94×10^5
	6	298.67	303.50	4.83	705	6.4693	6.4345	3.78×10^5	3.79×10^5	4.00×10^5
ALU-CPA-3B-1	1	297.47	296.06	-1.41	131	7.4735	7.4180	-0.96×10^5	-9.94×10^4	-0.78×10^5
	2	297.86	296.95	-0.91	205	7.4180	7.3941	-0.62×10^5	-6.62×10^4	-0.45×10^5
	3	298.10	297.66	-0.44	252	7.3941	7.3734	-0.30×10^5	-3.29×10^4	-0.12×10^5
	4	298.92	300.42	1.50	423	7.3734	7.3435	1.03×10^5	1.01×10^5	1.22×10^5
	5	299.46	302.40	2.94	531	7.3435	7.3028	2.03×10^5	2.02×10^5	2.23×10^5
	6	298.60	303.23	4.63	641	7.3028	7.2859	3.20×10^5	3.20×10^5	3.41×10^5
	7	298.04	305.31	7.27	810	7.2859	7.2718	5.04×10^5	5.06×10^5	5.27×10^5

Note : 1. K(Calorimeter constant) = 503.7 Joules/ K

TABLE 18

ENTHALPY OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION
(ICE CALORIMETER)

Spec. and Drop No.	Spec. temp. K	Soak time at temp. min.	Initial weight of C. S. graphite drop cup g	Initial wt. of spec. g	Initial wt. of cup and spec. g	W_s Final wt. of spec. g	Spec. wt. loss %	Total mercury displacement cm^3	Mercury displacement due to cup cm^3	Mercury displacement due to spec cm^3	Enthalpy above 273 K reference Joules/Kg
AL-CPI-2B-1											
Drop 1	810	15	3.5099	5.4016	8.8518	5.3419	1.11	1.50	0.72	0.78	5.37×10^5
Drop 2	1105	15	3.5099	5.3419	8.8173	5.3074	0.65	2.33	1.13	1.20	8.31×10^5
Drop 3	1369	15	3.5099	5.3074	8.7995	5.2896	0.34	3.32	1.50	1.82	1.26×10^6
Drop 4	1120	15	3.5099	5.2896	8.7822	5.2723	0.33	2.39	1.14	1.25	8.71×10^5
Drop 5	1653	15	3.2193	5.3861	8.5840	5.3647	0.40	4.31	1.93	2.38	1.63×10^6
Drop 6	1933	15	3.2193	5.3647	8.5562	5.3369[3]	0.52	5.17	2.30	2.87	1.98×10^6
AL-CPI-2B-2											
Drop 1	810	15	3.5172	5.2773	8.7673	5.2501	0.52	1.36	0.72	0.64	4.45×10^5
Drop 2	1270	15	3.2160	5.2751	8.4856	5.2696	0.01	2.93	1.36	1.57	1.10×10^6
Drop 3	983	15	3.2160	5.2696	8.4839	5.2679	0.00	1.94	0.96	0.98	6.84×10^5
Drop 4	1539	15	3.2160	5.2679	8.4705	5.2545	0.03	3.73	1.74	1.99	1.39×10^6
Drop 5	1811	15	3.2160	5.2545	8.4478	5.2318[4]	0.04	4.73	2.12	2.61	1.83×10^6

Notes:

1. Enthalpy calculations based on value of 3.680×10^3 Joules/cm^3 of ice melted.
2. Final weight used for calculation of enthalpy.
3. Final weight = 5.3727 g
4. Final weight = 5.2655 g

TABLE 19

ENTHALPY OF MULLITE FIBER RIGIDIZED INSULATION
(ADIABATIC CALORIMETER)

Specimen	Drop No.	Initial cup temp. K	Final cup temp. K	Change in cup temp. K	Initial temp. of sample K	Initial wt. of sample g	W_s final wt. of sample g	Enthalpy [1] $h = \frac{k}{W_s}(t_2 - t_1)$ Joules/Kg	Enthalpy Joules/Kg above 303 K	Enthalpy Joules/Kg above 273 K
MUL-CPA-2A-1	1	298.89	297.53	-1.36	148	7.4057	7.3635	-0.93×10^5	-9.56×10^4	-0.73×10^5
	2	298.58	297.62	-0.96	227	7.3635	7.3233	-0.66×10^5	-6.06×10^4	-0.38×10^5
	3	298.81	299.65	0.84	366	7.3233	7.2785	0.58×10^5	0.56×10^4	0.79×10^5
	4	299.07	301.34	2.27	480	7.2785	7.2426	1.58×10^5	1.57×10^5	1.80×10^5
	5	298.14	302.26	4.12	591	7.2426	7.1970	2.88×10^5	2.88×10^5	3.11×10^5
	6	298.85	304.50	5.65	704	7.1970	7.1835	3.96×10^5	3.98×10^5	4.21×10^5
MUL-CPA-2A-2	1	298.02	296.61	-1.41	123	6.9235	6.8823	-1.03×10^5	-1.07×10^5	-0.84×10^5
	2	297.98	297.08	-0.90	196	6.8823	6.8721	-0.66×10^5	-7.04×10^4	-0.47×10^5
	3	297.99	297.55	-0.44	250	6.8721	6.8905	-0.32×10^5	-3.58×10^4	-0.126×10^5
	4	297.98	299.54	1.56	418	6.8905	6.8085	1.15×10^5	1.12×10^5	1.35×10^5
	5	299.18	302.04	2.86	530	6.8085	6.7869	2.12×10^5	2.12×10^5	2.35×10^5
	6	298.64	303.01	4.37	643	6.7869	6.7724	3.25×10^5	3.25×10^5	3.48×10^5
	7	297.92	304.96	7.04	816	6.7724	6.7583	5.25×10^5	5.27×10^5	5.50×10^5

Note : 1. K(Calorimeter constant) = 503.7 Joules/ K

TABLE 20

ENTHALPY OF MULLITE FIBER RIGIDIZED INSULATION (ICE CALORIMETER)

Spec. and Drop No.	Spec. temp. K	Soak time at temp. min.	Initial weight of C. S. graphite drop cup g	Initial wt. of spec. g	Initial wt. of cup and spec. g	Final wt. of spec. g	Spec. wt. loss %	Total mercury displacement cm^3	Mercury displacement due to cup cm^3	Mercury displacement cue to spec. cm^3	Enthalpy above 273 K reference Joules/kg
MUL-CPI-2A-1											
Drop 1	810	15	3.4321	5.3671	8.7585	5.3264	0.76	1.47	0.72	0.75	5.18×10^5
Drop 2	1096	15	3.4321	5.3264	8.6861	5.2540	1.36	2.30	1.12	1.18	8.25×10^5
Drop 3	1368	15	3.4321	5.2540	8.6445	5.2124	0.79	3.33	1.50	1.83	1.29×10^6
Drop 4	1120	15	3.4321	5.2124	8.6361	5.2040	0.16	2.41	1.14	1.27	8.97×10^5
Drop 5	1653	15	3.2106	5.3525	8.5385	5.3279	0.46	4.14	1.90	2.24	1.55×10^6
Drop 6	1928	15	3.2106	5.3279	8.2328	5.0222[3]	5.74	5.08	2.29	2.79	2.04×10^6
MUL-CPI-2A-2											
Drop 1	811	15	3.3988	6.5471	9.9141	6.5153	0.49	1.52	0.72	0.80	4.51×10^5
Drop 2	1274	15	3.2122	6.5471	9.7530	6.5436	0.05	3.30	1.37	1.93	1.08×10^6
Drop 3	981	15	3.2122	6.5436	9.7525	6.5431	0.01	2.17	0.96	1.21	6.80×10^5
Drop 4	1566	15	3.2122	6.5431	9.7388	6.5294	0.21	4.34	1.78	2.56	1.44×10^6
Drop 5	1819	15	3.2122	6.5294	9.6668	6.4574[4]	1.10	5.40	2.13	3.27	1.81×10^6

Notes:

1. Enthalpy calculations based on value of 3.680×10^3 Joules/cm^3 of ice melted.
2. Final weight used for calculations of enthalpy.
3. Final weight = 5.0845 g
4. Final weight = 6.4914 g

TABLE 21

ENTHALPY OF LI-1500 RIGIDIZED INSULATION
(ADIABATIC CALORIMETER)

Specimen	Drop No.	Initial cup temp. K	Final cup temp. K	Change in cup temp. K	Initial temp. wt. sample K	Initial wt. of sample g	W_s Final wt. of sample g	Enthalpy[1] $h = \frac{k}{W_s}(t_2-t_1)$ Joules/kg	Enthalpy Joules/kg above 303 K	Enthalpy Joules/kg above 273 K
LI-1500-CPA-1	1	297.89	296.39	-1.50	149	8.3495	8.3006	-0.91×10^5	-9.47×10^4	-0.73×10^5
	2	297.79	297.02	-0.77	227	8.3006	8.2903	-0.47×10^5	-5.02×10^4	-0.281×10^5
	3	298.62	299.53	0.91	364	8.2903	8.2581	0.56×10^5	5.28×10^4	0.749×10^5
	4	299.58	302.01	2.43	484	8.2581	8.2404	1.49×10^5	1.48×10^5	1.70×10^5
	5	297.44	301.72	4.28	594	8.2404	8.2317	2.62×10^5	2.61×10^5	2.83×10^5
	6	298.76	304.85	6.09	701	8.2317	8.2238	3.73×10^5	3.75×10^5	3.97×10^5
LI-1500-CPA-2	1	297.94	296.39	-1.55	138	8.3980	8.3755	-0.93×10^5	-9.66×10^4	-0.745×10^5
	2	298.64	297.57	-1.07	203	8.3755	8.3521	-0.65×10^5	-6.84×10^4	-0.463×10^5
	3	298.19	297.66	-0.53	251	8.3521	8.3816	-0.32×10^5	-3.54×10^4	-0.133×10^5
	4	299.04	300.73	1.69	419	8.3816	8.2734	1.03×10^5	1.01×10^5	1.23×10^5
	5	299.10	302.41	3.31	530	8.2734	8.2626	2.02×10^5	2.02×10^5	2.24×10^5
	6	297.90	303.03	5.13	643	8.2626	8.2533	3.13×10^5	3.12×10^5	3.34×10^5
	7	298.11	305.86	7.75	811	8.2533	8.2445	4.73×10^5	4.77×10^5	4.99×10^5

Note : 1. K(Calorimeter constant) = 503.7 Joules/ K

TABLE 22

ENTHALPY OF LI-1500 RIGIDIZED INSULATION
(ICE CALORIMETER)

Spec. & Run No.	Spec. temp. K	Soak Time at temp min.	Initial weight of C. S. graphite drop cup g	Initial wt. of spec. g	Initial wt. of cup & spec. g	Final wt. of spec. g	Spec. wt. loss %	Total mercury displacement cm^3	Mercury displacement due to cup cm^3	Mercury displacement due to spec. cm^3	Enthalpy above 273K reference Joules/Kg
LI-1500-CPI-1											
Drop 1	811	15	3.497	3.9685	7.4290	3.9320	0.91	1.14	0.72	0.42	3.93×10^5
Drop 2	1109	15	3.2097	3.9582[2]	7.1649	3.9552	0.07	1.96	1.13	0.83	7.71×10^5
Drop 3	1381	15	3.2097	3.9552	7.1569	3.9472	0.20	2.81	1.52	1.29	12.0×10^5
Drop 4	1669	15	3.2097	3.9472	7.1451	3.9354	0.29	3.65	1.92	1.73	16.2×10^5
Drop 5	1928	15	3.2097	3.9354	7.0606	3.8509	2.14	4.49	2.29	2.20	21.0×10^5
Drop 6	1928	15	3.2097[4]	3.8509	7.0230	3.8133[5]	0.97	4.25	2.29	1.96	18.9×10^5
LI-1500-CPI-2											
Drop 1	811	15	3.5030	3.6266	7.0986	3.5956	0.85	1.05	0.72	0.33	3.37×10^5
Drop 2	1272	15	3.2142	3.8613[2]	7.0690	3.8548	0.16	2.50	1.35	1.15	11.0×10^5
Drop 3	989	15	3.2142	3.8548	7.0653	3.8511	0.09	1.39	0.97	0.42	4.01×10^5
Drop 4	985	15	3.2142	3.8511	7.0613	3.8471	0.10	1.59	0.96	0.63	6.02×10^5
Drop 5	1566	15	3.2142	3.8471	7.0505	3.8363	0.28	3.30	1.78	1.52	14.6×10^5
Drop 6	1830	15	3.2142	3.8363	6.9922	3.7780	1.51	3.94	2.16	1.78	17.3×10^5
LI-1500-CPI-3											
Drop 1	815	15	3.3256	4.1903	7.4089	4.1833	0.16	1.24	0.72	0.52	4.57×10^5
Drop 2	1655	15	3.3256	4.1833	7.3735	4.1479	0.84	3.61	1.90	1.71	15.2×10^5

Notes:

1. Enthalpy calculations based on value of 3.680×10^3 Joules/cm^3 of ice melted.
2. Specimen repacked and specimen weight redetermined.
3. Final weight used for calculations of enthalpy.
4. Final weight = 3.1764 gm
5. Final weight = 3.8469 gm

TABLE 23

THERMAL EXPANSION OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE QUARTZ DILATOMETER

Specimen	Time	Specimen temperatures - K				Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit elongation correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
		Top	Middle	Bottom	Average				
Spec AL-QEXP-1B-W Run 5317-20-14 Density: 0.2468 gm/cm^3		Initial Length: 7.622 cm Final Length: 7.587 cm				Initial Weight: 2.3417 g Final Weight: 2.3019 g			
	11:32am	300	300	300	300	0.00	0.00	0.00	0.00
	12:48pm	116	121	136	130	-3.28	-0.43	0.03	-0.40
	12:53pm	140	150	159	150	-2.82	-0.37	0.01	-0.36
	1:00pm	166	171	176	171	-2.57	-0.34	0.00	-0.34
	1:14pm	195	200	203	199	-2.01	-0.26	-0.01	-0.27
	2:08pm	225	227	229	227	-1.68	-0.22	-0.02	-0.23
	2:50pm	254	255	257	255	-1.19	-0.16	-0.01	-0.17
	4:03pm	298	298	298	298	-0.23	-0.03	0.00	-0.03
	9:08am	364	364	364	364	1.37	0.18	0.02	0.20
	9:55am	430	431	430	431	3.94	0.52	0.05	0.57
	10:25am	478	478	478	478	7.82	1.03	0.08	1.10
	10:53am	533	534	533	533	11.48	1.51	0.11	1.61
	11:38am	589	589	588	589	13.44	1.76	0.11	1.87
	12:13pm	659	659	658	659	14.25	1.87	0.18	2.05
	12:38pm	698	699	792	698	17.02	2.23	0.21	2.44
	1:15pm	755	755	754	755	18.87	2.48	0.24	2.72
	1:37pm	821	821	818	820	21.08	2.77	0.28	3.05
	1:53pm	874	874	871	873	22.25	2.92	0.51	3.43
	2:15pm	918	920	917	919	22.83	2.99	0.54	3.53
	2:37pm	979	980	975	978	23.52	3.08	0.38	3.46
	2:55pm	1033	1034	1028	1031	23.77	3.12	0.41	3.53
	3:15pm	1089	1088	1083	1086	22.89	3.00	0.44	3.44
	7:25am	300	300	300	300	-11.51	-1.51	0.00	-1.51

TABLE 24

THERMAL EXPANSION OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE QUARTZ DILATOMETER

Specimen	Time	Specimen temperatures - K				Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit elongation correction for dilatometer motion 10^{-3}cm/cm	Corrected specimen unit elongation 10^{-3}cm/cm
		Top	Middle	Bottom	Average				
Spec AL-QEXP-2B-W Run 5317-26-15 Density: 0.2321 gm/cm^3		Initial Length: 7.617 cm Final Length: 7.565 cm				Initial Weight: 2.1082 g Final Weight: 2.0661 g			
	12:15	299	299	299	299	0.00	0.00	0.00	0.00
	12:21	84	78	78	80	-4.14	-0.54	0.079	-0.46
	12:34	148	149	151	149	-2.97	-0.39	0.010	-0.38
	12:49	200	202	203	202	-2.24	-0.29	-0.011	-0.30
	1:11	230	230	231	230	-1.65	-0.22	-0.015	-0.23
	1:58	255	255	255	255	-1.17	-0.15	-0.012	-0.16
	3:00	299	299	299	299	0.00	0.00	0.00	0.00
	8:20	343	343	344	344	0.99	0.13	0.01	0.14
	8:45	394	394	395	394	2.51	0.33	0.03	0.36
	9:10	449	450	450	450	4.27	0.56	0.06	0.62
	9:35	505	506	506	506	7.54	0.99	0.09	1.08
	10:00	561	563	564	563	9.47	1.24	0.12	1.36
	10:25	615	616	618	616	11.30	1.48	0.15	1.63
	11:00	668	671	673	670	12.88	1.69	0.19	1.88
	11:20	726	730	732	729	14.91	1.96	0.22	2.18
	11:50	779	783	786	783	16.48	2.16	0.25	2.41
	12:30	835	840	844	840	18.34	2.41	0.29	2.70
	1:00	889	895	900	895	19.15	2.51	0.32	2.83
	1:25	944	951	956	950	19.30	2.53	0.35	2.88
	1:50	1006	1015	1021	1014	19.15	2.51	0.39	2.90
	2:20	1052	1061	1068	1060	16.94	2.22	0.42	2.64
	7:35	300	300	300	300	-12.09	-1.59	0.00	-1.59

TABLE 25

THERMAL EXPANSION OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-1B-W	294	9:20	0.00	0.00	0.00	0.00
Run 5317-44-127	571	10:05	8.28	1.09	0.33	1.42
Initial length:	847	10:50	8.43	1.11	0.88	1.99
7.603 cm	1122	11:35	8.26	1.09	1.60	2.69
Final length:	1272	12:20	7.32	0.96	2.03	2.99
5.490 cm	1483	1:20	-1069.21	-140.60	2.77	-137.88
Initial weight:						
2.3360 g						
Final weight:						
2.3150 g						
Density: 0.2553 gm/cm^3						

Note: Specimen length reduced below range of dial gage

TABLE 26

THERMAL EXPANSION OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-2B-W	294	8:30	0.00	0.00	0.00	0.00
Run 5317-47-115R	562	9:15	8.84	1.16	0.01	1.17
Initial length:	816	10:00	14.81	1.19	0.29	2.24
7.601 cm	1050	10:45	18.42	2.42	0.70	3.12
Final length:	1238	11:30	16.03	2.11	1.13	3.24
6.490 cm	1433	11:50	-237.24	-31.54	1.60	-29.94
Initial weight:	1469	12:10	-691.95	-91.02	1.61	-89.41
2.2059 g	1472	12:25	-938.33	-123.42	1.70	-121.72
Final weight:	1472	12:40	-1116.13	-146.81	1.70	-145.11
2.1960 g						
Density: 0.2323 gm/cm^3						

Note: Specimen length reduced below range of dial gage

TABLE 27

THERMAL EXPANSION OF ALUMINA-SILICA-CHROMIA RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION MEASURED IN GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-1B-A	294	8:55	0.00	0.00	0.00	0.00
Run 5317-42-127	540	9:35	21.34	2.84	0.30	3.14
Initial length:	833	10:20	29.21	3.88	0.83	4.71
7.524 cm	1134	11:05	2.26	0.30	1.63	1.93
Final length:	1272	11:45	-38.35	-5.09	2.01	-3.08
2.452 cm	1455	12:30	-958.34	-127.34	2.67	-124.67
Initial weight:	1700	1:00	(See Note)			
2.1010 g						
Final weight:						
2.0743 g						
Density: 0.2276 gm/cm^3						

Note: Specimen length reduced below range of dial gage

TABLE 28

SUMMARY OF DIMENSIONAL CHANGES OF ALUMINA-SILICA-CHROMIA, MULLITE FIBER AND LI-1500 AFTER THERMAL EXPOSURES

Material and Spec No.	Direction	Max. Exposure Temp. K	Length			Dia. or "a" (W/G) Dimension			"b" Dimension		
			Initial cm	Final cm	Unit Change 10^{-3} cm/cm	Initial cm	Final cm	Unit Change 10^{-3} cm/cm	Initial cm	Final cm	Unit Change 10^{-3} cm/cm
Alumina-Silica-Chromia Rigidized Insulation											
AL-GEXP-1B-A	Across	1669	7.524	2.452	-674				1.252	1.003	-199
AL-GEXP-1B-W	With	1497	7.603	5.490	-278	1.240	1.207	-27			
AL-GEXP-2B-W	With	1472	7.601	6.490	-146	1.264	1.215	-38			
AL-Blank-2B (Heat Soak)	With	1861	7.788	3.642	-532	1.612	0.950	-410	1.416	0.787	-444
Mullite Fiber Rigidized Insulation											
MUL-GEXP-1C-A	Across	1697	7.545	6.425	-148				1.257	1.235	-18
MUL-GEXP-1C-W	With	1722	7.609	6.394	-160	1.245	1.232	-10			
MUL-GEXP-2A-W	With	1736	7.582	6.296	-170	1.248	1.246	-1.6			
MUL-GEXP-2A-W2	With	1744	7.614	6.787	-109	1.254	1.264	+7.7			
MUL-1C-W2 (Heat Soak)	With	1853	8.788	8.717	-8	1.462*	1.381*	-55*	1.616*	1.627*	+7.2*
MUL-W-3 (Heat Soak)	With	1864	8.804	8.715	-10	1.458*	1.354*	-7*	1.614*	1.617*	+2.4*
MUL-1C-W2 (Exp. after heat soak)	With	1816	7.610	6.741	-114	1.250	1.239	-9.6			
MUL-W-3 (Exp. after heat soak)	With	1811	7.558	6.750	-107	1.212	1.237	20.8			
LI-1500 Rigidized Insulation											
LI-1500-GEXP-A-1	Across	1922	7.656	6.446	-158				1.270	1.220	-39
LI-1500-GEXP-W-1	With	1458	7.616	6.982	-83	1.274	1.207	-53			
LI-1500-GEXP-W-2	With	1883	7.615	6.976	-84	1.270	1.184	-68			
LI-1500-Blank (Heat Soak)	With	1864	7.247	6.918	-45	1.625	1.557	-42	1.355	1.324	-23

All Heat Soak Specimens were Unloaded.

* With and Across Lamina Orientations for These Measurements are Unknown.

TABLE 29

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE QUARTZ DILATOMETER

Specimen	Time	Specimen temperatures - K				Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit elongation correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
		Top	Middle	Bottom	Average				
Spec MUL-QEXP-1C-W Run 5317-22-15 Density: 0.2247 gm/cm^3		Initial Length: 7.620 cm Final Length: 7.591 cm				Initial Weight: 2.0720 g Final Weight: 2.0431 g			
	11:35am	300	300	300	300	0.00	0.00	0.00	0.00
	12:30pm	98	100	109	103	-2.62	-0.34	0.05	-0.29
	12:40pm	150	151	151	151	-1.88	-0.25	0.01	-0.24
	12:56pm	174	175	180	176	-1.60	-0.21	-0.00	-0.21
	1:09pm	196	196	197	196	-1.42	-0.19	-0.01	-0.20
	2:05pm	231	231	233	231	-0.89	-0.12	-0.02	-0.14
	2:41pm	256	256	257	256	-0.66	-0.09	-0.01	-0.10
	6:45pm	298	298	298	298	0.23	0.03	0.00	0.03
	9:30am	363	363	363	363	1.63	0.21	0.02	0.24
	10:10am	424	425	425	425	3.84	0.50	0.05	0.55
	10:41am	481	481	481	481	7.09	0.93	0.08	1.00
	11:06am	534	533	532	533	8.66	1.14	0.10	1.24
	11:35am	592	592	590	591	10.69	1.40	0.11	1.51
	12:14pm	653	653	651	653	12.62	1.66	0.18	1.83
	12:41pm	702	702	699	701	14.12	1.85	0.21	2.06
	1:05pm	759	759	758	758	16.28	2.14	0.24	2.38
	1:40pm	816	816	812	815	17.81	2.34	0.27	2.61
	2:07pm	866	865	860	864	19.35	2.54	0.30	2.84
	2:40pm	922	920	915	919	20.75	2.72	0.34	3.06
	3:07pm	993	991	984	989	22.33	2.93	0.38	3.31
	3:25pm	1036	1034	1023	1032	23.22	3.05	0.40	3.45
	3:50pm	1108	1103	1095	1102	24.10	3.16	0.45	3.61
	7:28am	300	300	300	300	-4.57	-0.60	0.00	-0.60

TABLE 30

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION
MEASURED IN THE QUARTZ DILATOMETER

Specimen	Time	Specimen temperatures - K				Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit elongation correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
		Top	Middle	Bottom	Average				
Spec MUL-QEXP-2A-W Run 5317-24-14 Density: 0.2764 gm/cm³		Initial Length: 7.615 cm Final Length: 7.606 cm				Initial Weight: 2.4780 g Final Weight: 2.4617 g			
	12:13	299	299	299	299	0.00	0.00	0.00	0.00
	12:25	126	119	115	120	-2.39	-0.31	0.033	-0.28
	12:35	151	145	144	146	-2.08	-0.27	-0.011	-0.26
	12:46	199	199	199	199	-1.42	-0.19	-0.010	-0.20
	2:03	256	256	256	256	-0.81	-0.11	-0.011	-0.12
	7:45	299	299	299	299	-0.08	-0.01	0.00	-0.01
	8:20	339	340	341	340	0.89	0.12	0.01	0.13
	8:45	394	394	395	395	2.39	0.31	0.04	0.35
	9:10	451	451	451	451	5.16	0.68	0.06	0.74
	9:35	505	506	506	506	7.44	0.98	0.09	1.07
	10:05	561	561	562	561	9.22	1.21	0.12	1.33
	10:30	619	620	620	620	11.07	1.45	0.16	1.61
	10:55	672	672	673	672	12.80	1.68	0.19	1.87
	11:20	726	728	728	728	14.50	1.90	0.22	2.12
	11:50	781	782	782	782	16.23	2.13	0.26	2.39
	12:30	841	843	843	843	18.26	2.40	0.29	2.69
	1:00	894	895	895	894	19.96	2.62	0.32	2.94
	1:20	951	953	955	953	21.72	2.85	0.36	3.21
	1:45	1006	1008	1009	1008	23.47	3.08	0.39	3.47
	2:20	1058	1061	1063	1061	24.92	3.27	0.43	3.70
	7:35	300	300	300	300	-2.21	-0.29	0.00	-0.29

TABLE 31

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-1C-W	294	9:15	0.00	0.00	0.00	0.00
Run 5317-45-115R	554	10:15	9.78	1.29	0.01	1.30
Initial length:	826	11:10	16.15	2.12	0.30	2.42
7.609 cm	1102	12:15	22.02	2.89	0.81	3.70
Final length:	1224	1:15	24.38	3.20	1.10	4.30
6.394 cm	1466	2:10	27.69	3.64	1.70	5.34
Initial weight:	1697	2:55	(See Note)			
2.0798 g						
Final weight:						
2.0471 g						
Density: 0.2255 gm/cm^3						

Note: Specimen length reduced below readable range of dial gage

TABLE 32

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-2A-W-2	297	9:20	0.00	0.00	0.00	0.00
Run 5317-52-127	569	10:00	7.44	0.98	0.33	1.31
Initial length:	805	10:35	12.37	1.62	0.77	2.39
7.614 cm	1185	11:15	20.47	2.69	1.76	4.45
Final length:	1389	11:50	24.49	3.22	2.45	5.67
6.787 cm	1508	12:25	24.97	3.28	2.86	6.14
Initial weight:	1622	1:00	-2.51	-0.33	3.24	2.91
2.5043 g	1744	1:23	(See Note)			
Final weight:						
2.4556 g						
Density: 0.2663 gm/cm^3						

Note: Specimen length reduced below dial range

TABLE 33

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	orrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-2A-W	294	8:45	0.00	0.00	0.00	0.00
Run 5317-46-127	564	9:30	3.73	0.49	0.33	0.82
Initial length:	823	10:15	3.63	0.48	0.80	1.28
7.582 cm	1138	11:00	3.43	0.45	1.63	2.08
Final length:	1287	11:50	3.20	0.42	2.10	2.52
6.296 cm	1497	12:35	3.07	0.41	2.80	3.21
Initial weight:	1586	12:40	3.07	0.41	3.10	3.51
2.5142 g	1622	12:41	2.44	0.32	3.23	3.55
Final weight:	1641	12:42	-5.18	-0.68	3.30	2.62
2.4325 g	1664	12:44	-22.96	-3.03	3.36	0.33
Density: 0.2722 gm/cm^3	1675	12:46	-48.36	-6.38	3.42	-2.96
	1680	12:48	-73.76	-9.73	3.42	-6.31
	1703	12:50	-276.96	-36.53	3.51	-33.02
	1736	1:15	(See Note)			

Note: Specimen length reduced below readable range of dial gage

TABLE 34

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION AFTER HEAT TREATMENT AT 1850K (MEASURED IN GRAPHITE DILATOMETER)

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec MUL-1C-W-2	297	8:00	0.00	0.00	0.00	0.00
Run 5317-57-129	537	8:40	3.76	0.49	0.30	0.79
Initial length:	818	9:20	7.54	0.99	0.87	1.86
7.610 cm	1210	10:00	10.46	1.38	2.02	3.40
Final length:	1372	10:40	10.85	1.43	2.57	4.00
6.741 cm	1514	11:20	9.17	1.20	3.00	4.20
Initial weight:	1622	12:00	-2.79	-0.37	3.38	3.01
2.4645 g	1725	12:40	-141.48	-18.59	3.72	-14.87
Final weight:	1816	1:00	----	See note		----
2.3794 g						
Density: 0.2637 gm/cm^3						

Note: Specimen length reduced below readable range of dial gage.

TABLE 35

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION AFTER HEAT TREATMENT AT 1850K (MEASURED IN GRAPHITE DILATOMETER)

Specimen and Run No.	Temp K	Time	Observed total elongation 10^{-3}cm	Observed unit elongation 10^{-3}cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec MUL-W-3	296	7:40	0.00	0.00	0.00	0.00
Run 5317-56-127	565	8:20	4.55	0.60	0.34	0.94
Initial length:	821	9:05	7.93	1.05	0.79	1.84
7.558 cm	1160	9:50	10.24	1.35	1.70	3.05
Final length:	1330	10:35	9.63	1.27	2.23	3.50
6.750 cm	1494	11:15	7.93	1.05	2.80	3.85
Initial weight:	1633	12:00	-7.36	-0.97	3.29	2.32
2.4104 g	1733	12:40	-193.50	-25.60	3.64	-21.96
Final weight:	1811	1:00		See note		
2.3540 g						
Density: 0.2760 gm/cm^3						

Note: Specimen length reduced below readable range of dial gage.

TABLE 36

THERMAL EXPANSION OF MULLITE FIBER RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION MEASURED IN GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3}cm	Observed unit elongation 10^{-3}cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-1C-A	294	8:30	0.00	0.00	0.00	0.00
Run 5317-41-115R	575	9:15	11.61	1.54	0.02	1.56
Initial length:	841	10:00	18.42	2.44	0.31	2.75
7.545 cm	1146	10:45	23.16	3.07	0.92	3.99
Final length:	1294	11:30	21.74	2.88	1.26	4.14
6.425 cm	1497	12:15	11.51	1.52	1.76	3.28
Initial weight:	1697	12:45	(See Note)			
2.1233 g						
Final weight:						
2.0980 g						
Density: 0.2275 gm/cm^3						

Note: Specimen length reduced below range of dial gage

TABLE 37

THERMAL EXPANSION OF LI-1500 RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE QUARTZ DILATOMETER

Specimen	Time	Specimen temperatures - K				Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit elongation correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
		Top	Middle	Bottom	Average				
Spec LI-1500-QEXP W-1		Initial Length: 7.615 cm				Initial Weight: 2.7123 g			
Run 5317-34-15		Final Length: 7.614 cm				Final Weight: 2.6934 g			
Density: 0.2822 gm/cm³	1:20pm	301	301	301	301	0.00	0.00	0.00	0.00
	1:35	143	139	144	142	-0.58	-0.08	0.01	-0.07
	1:40	153	149	151	151	-0.51	-0.07	0.01	-0.06
	1:45	172	171	172	172	-0.46	-0.06	0.00	-0.06
	2:10	228	226	227	227	-0.25	-0.03	0.02	-0.05
	8:40am	301	301	301	301	0.00	0.00	0.00	0.00
	10:25	366	367	367	367	0.15	0.02	0.02	0.04
	11:00	423	424	424	424	0.23	0.03	0.05	0.08
	11:25	476	478	478	478	0.23	0.03	0.07	0.10
	11:40	541	541	542	541	0.25	0.03	0.11	0.14
	12:03	589	590	590	590	0.30	0.04	0.14	0.18
	12:35	641	643	643	641	0.30	0.04	0.17	0.21
	1:00	698	701	701	699	0.33	0.04	0.20	0.25
	1:25	755	757	758	756	0.41	0.05	0.24	0.29
	1:45	814	815	813	814	0.81	0.11	0.27	0.38
	2:00	871	875	876	873	1.30	0.17	0.31	0.48
	2:20	916	920	921	919	1.35	0.18	0.34	0.52
	2:45	981	985	985	983	1.37	0.18	0.37	0.55
	3:00	1029	1035	1036	1033	1.50	0.20	0.40	0.60
	3:20	1082	1088	1089	1087	1.35	0.18	0.44	0.61
	8:02am	300	300	300	300	0.61	0.08	0.00	0.08

TABLE 38

THERMAL EXPANSION OF LI-1500 RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE QUARTZ DILATOMETER

Specimen	Time	Specimen temperatures - K				Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit elongation correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
		Top	Middle	Bottom	Average				
Spec LI-1500-QEXP W-2 Run 5317-32-14 Density: 0.2854 gm/cm^3		Initial Length: 7.616 cm Final Length: 7.613 cm				Initial Weight: 2.7267 g Final Weight: 2.7194 g			
	1:20pm	301	301	301	301	0.00	0.00	0.00	0.00
	1:35	118	113	112	114	-0.53	-0.07	0.04	-0.03
	1:50	147	145	144	145	-0.48	-0.06	0.01	-0.05
	2:10	200	200	200	200	-0.36	-0.05	-0.01	-0.06
	2:30	256	255	255	255	-0.18	-0.02	-0.01	-0.03
	8:42am	300	300	300	300	0.00	0.00	0.00	0.00
	9:45	339	339	339	339	-0.05	0.01	0.01	0.01
	10:51	394	395	395	395	0.05	0.01	0.04	0.04
	11:18	451	452	452	451	0.05	0.01	0.06	0.07
	11:45	506	509	509	508	0.08	0.01	0.09	0.10
	12:18pm	561	563	562	562	0.18	0.02	0.12	0.14
	12:38	619	620	620	619	0.23	0.03	0.16	0.19
	12:52	673	676	676	675	0.28	0.04	0.19	0.23
	1:05	726	729	728	727	0.41	0.05	0.22	0.28
	1:20	781	783	781	781	0.67	0.09	0.26	0.34
	1:32	835	838	838	836	1.22	0.16	0.29	0.45
	1:52	894	898	899	897	1.55	0.20	0.33	0.53
	2:15	954	959	959	956	1.63	0.21	0.36	0.58
	2:38	998	1004	1004	1003	1.57	0.21	0.39	0.60
	3:00	1059	1065	1066	1063	1.57	0.21	0.43	0.64
	8:00am	302	302	302	302	0.61	0.08	0.00	0.08

TABLE 39

THERMAL EXPANSION OF LI-1500 RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-W-1	294	9:15	0.00	0.00	0.00	0.00
Run 5317-40-127	546	10:00	-0.86	-0.11	0.30	0.19
Initial length:	811	10:45	-3.66	-0.48	0.79	0.31
7.616 cm	1095	11:30	-8.13	-1.07	1.52	0.45
Final length:	1310	12:15	-13.87	-1.82	2.15	0.33
6.982 cm	1458	1:00	-62.74	-8.24	2.67	-5.57
Initial weight:						
2.7266 g						
Final weight:						
2.7190 g						
Density: 0.2820 gm/cm^3						

Note: Specimens length reduced below range of dial gage

TABLE 40

THERMAL EXPANSION OF LI-1500 RIGIDIZED INSULATION IN THE WITH LAMINA DIRECTION MEASURED IN THE GRAPHITE DILATOMETER

Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-W-2	294	8:45	0.00	0.00	0.00	0.00
Run 5317-48-115R	563	9:30	0.48	0.06	0.01	0.07
Initial length:	815	10:15	-0.81	-0.11	0.29	0.18
7.615 cm	1091	11:00	-3.28	-0.43	0.80	0.37
Final length:	1271	12:15	-6.55	-0.86	1.21	0.35
6.976 cm	1447	12:45	-39.70	-5.21	1.67	-3.52
Initial weight:	1461	1:00	-118.44	-15.55	1.67	-13.88
2.7385 g	1697	1:45	-581.71	-76.38	2.32	-74.08
Final weight:	1883	2:30	-568.80	-74.68	2.82	-71.86
2.6372 g	294	7:35	-687.91	-90.32	0.00	-90.32
Density: 0.2850 gm/cm^3						

Note: Specimen length reduced below readable dial range

TABLE 41

THERMAL EXPANSION OF LI-1500 RIGIDIZED INSULATION IN THE ACROSS LAMINA DIRECTION MEASURED IN THE GRAPHITE DILATOMETER

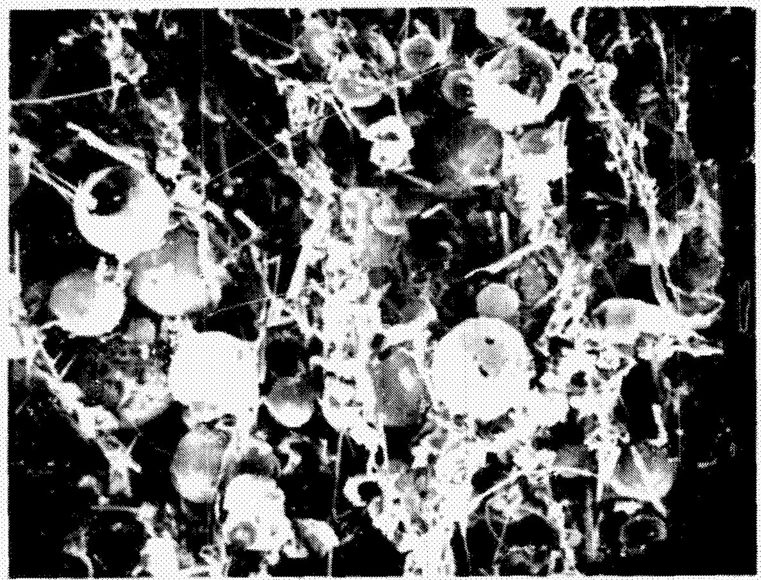
Specimen and Run No.	Temp. K	Time	Observed total elongation 10^{-3} cm	Observed unit elongation 10^{-3} cm/cm	Unit correction for dilatometer motion 10^{-3} cm/cm	Corrected specimen unit elongation 10^{-3} cm/cm
Spec GEXP-A-1	294	8:50	0.00	0.00	0.00	0.00
Run 5317-43-115R	572	9:30	1.78	0.23	0.02	0.25
Initial length:	846	10:15	1.70	0.22	0.32	0.54
7.656 cm	1116	11:00	-0.76	-0.10	0.85	0.75
Final length:	1263	11:45	-5.03	-0.66	1.20	0.54
6.446 cm	1491	12:30	-392.43	-51.25	1.75	-49.50
Initial weight:	1739	1:15	-1146.05	-149.66	2.42	-147.24
2.6348 g	1922	2:00	-1135.94	-148.34	2.95	-145.42
Final weight:						
2.4559 g						
Density: 0.2727 gm/cm^3						

Note: Specimen length reduced below range of dial gage

TABLE 42

SUMMARY OF RESULTS OF THERMAL ANALYSIS ON THREE RIGIDIZED INSULATORS

Material	Bulk density gm/cm^3	Estimated porosity %	Effective solid contribution to conductivity W/m-K	Estimated "true" conductivity of solid W/m-K	N Backscattering cross-section per unit volume m^{-1}	Estimated percent of available radiant heat flux being transmitted %
Alumina- Silica Chromia	0.22	89	0.048	0.66	4900	8
Mullite	0.24	89	0.043	0.55	3160	12.5
LI-1500	0.26	92	0.025	0.29	7130	5.5

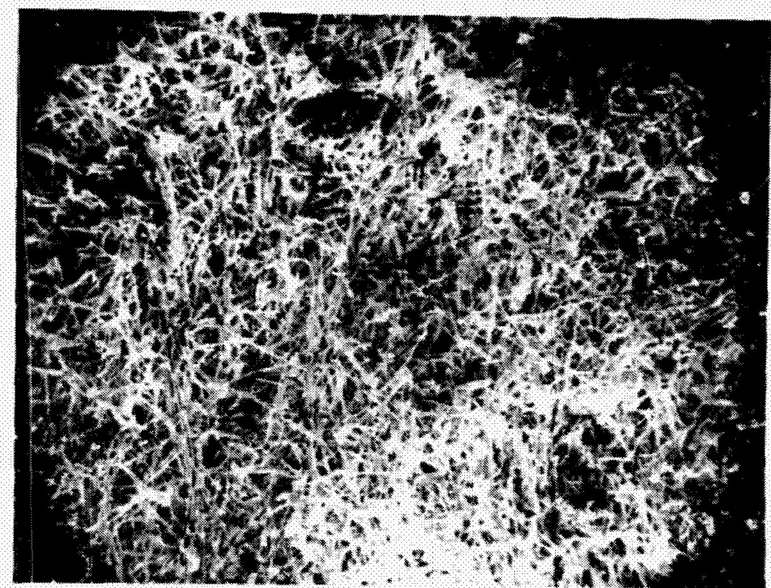


200 microns

Alumina-Silica-Chromia
(90X)

Mullite
(82X)

200
microns



LI-1500
(82X)

200
microns

Figure 1. Photomicrographs of Materials from Scanning Electron Microscope

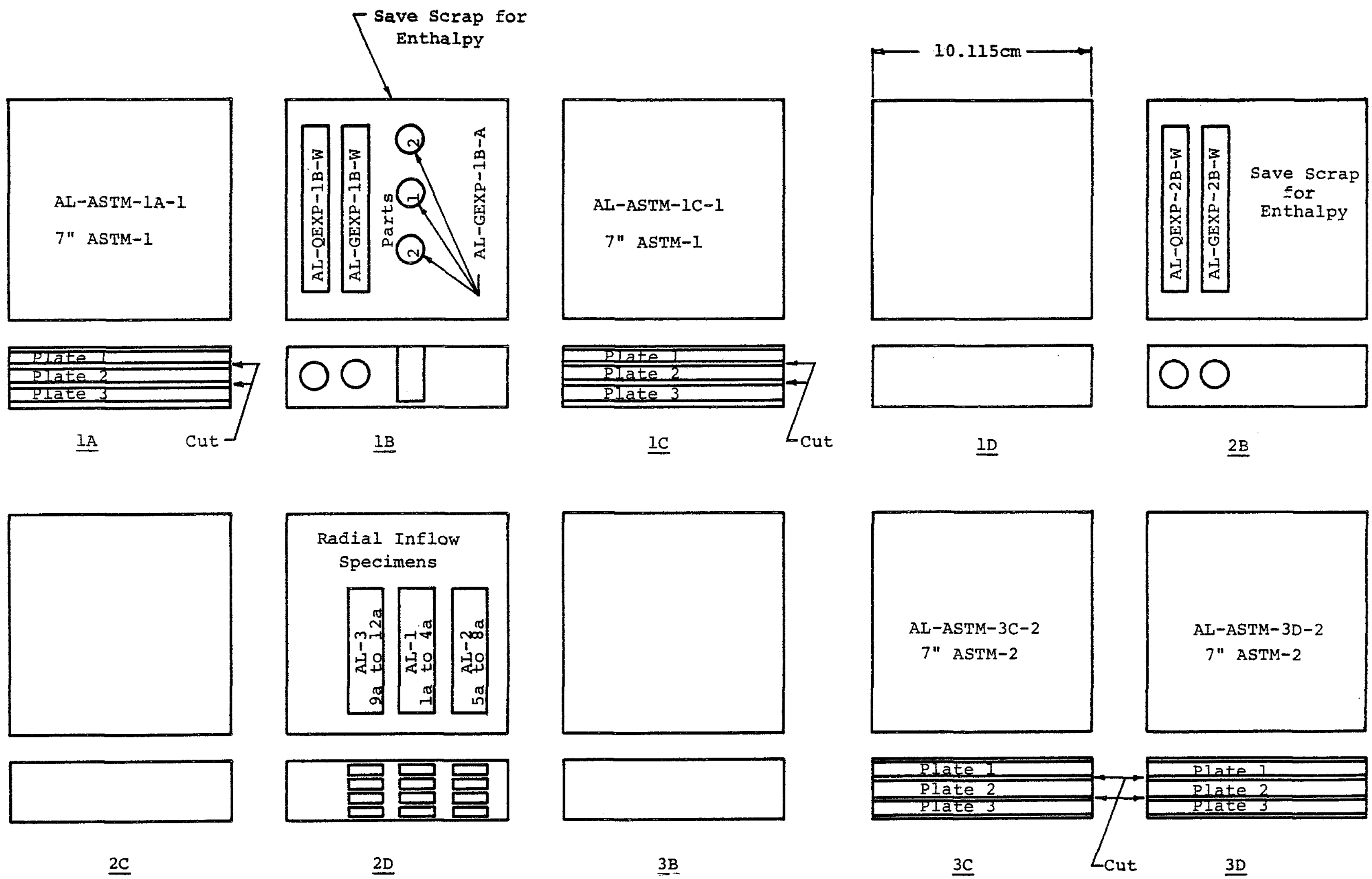


Figure 2. Cutting plans for Alumina-Silica-Chromia Rigidized Insulation

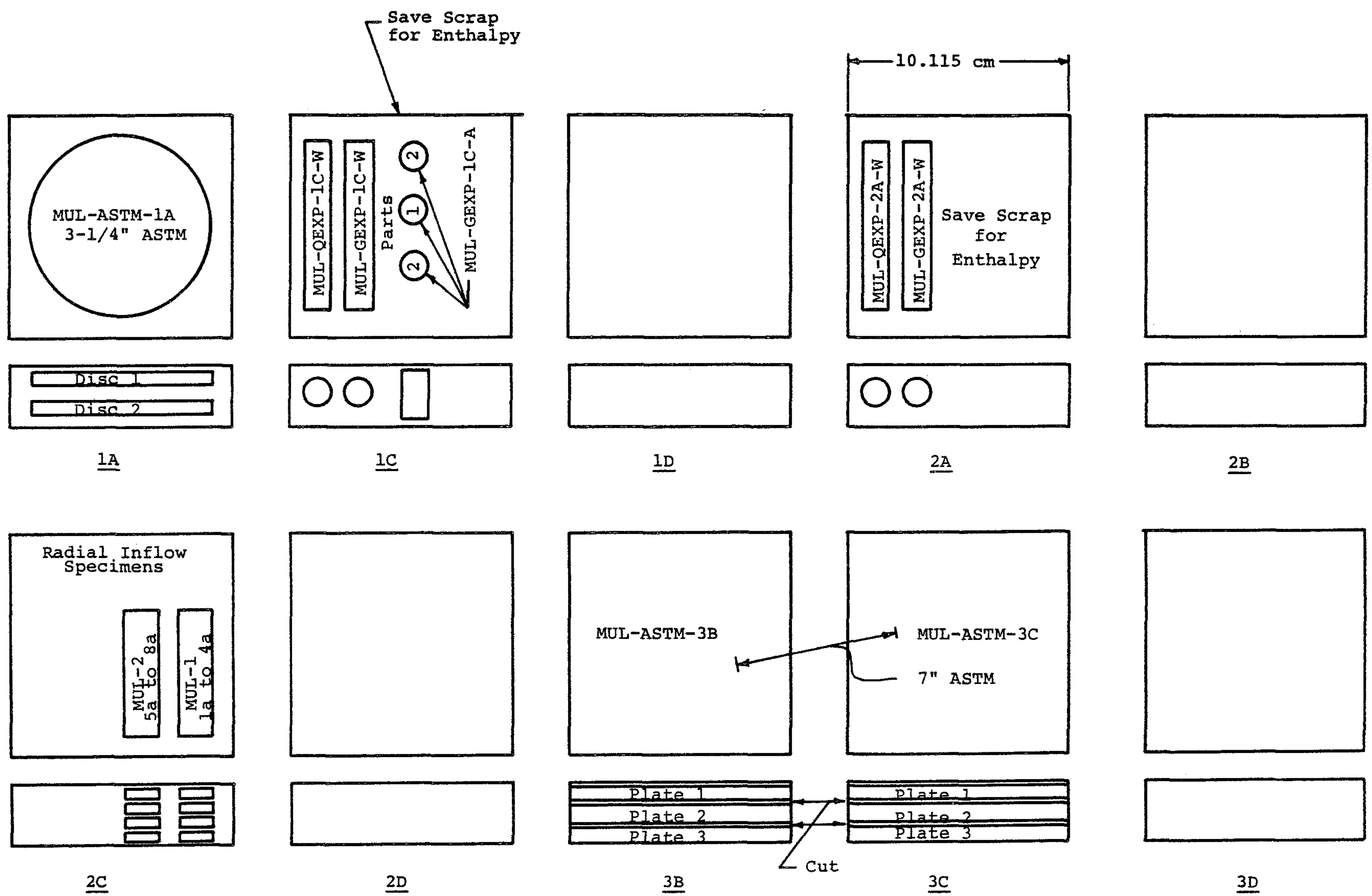


Figure 3. Cutting plans for Mullite Fiber Rigidized Insulation

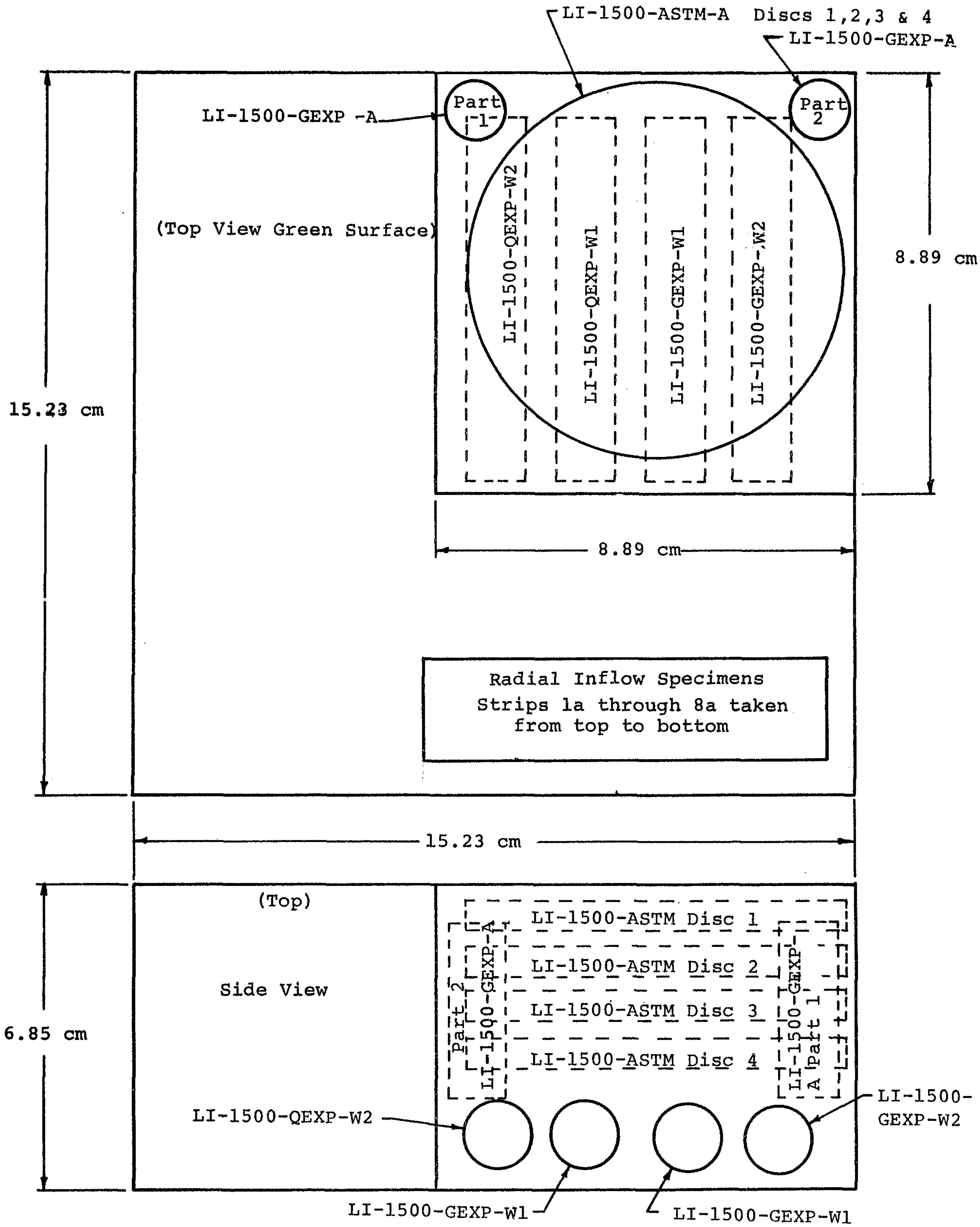


Figure 4. Cutting plan for LI-1500 Rigidized Insulation

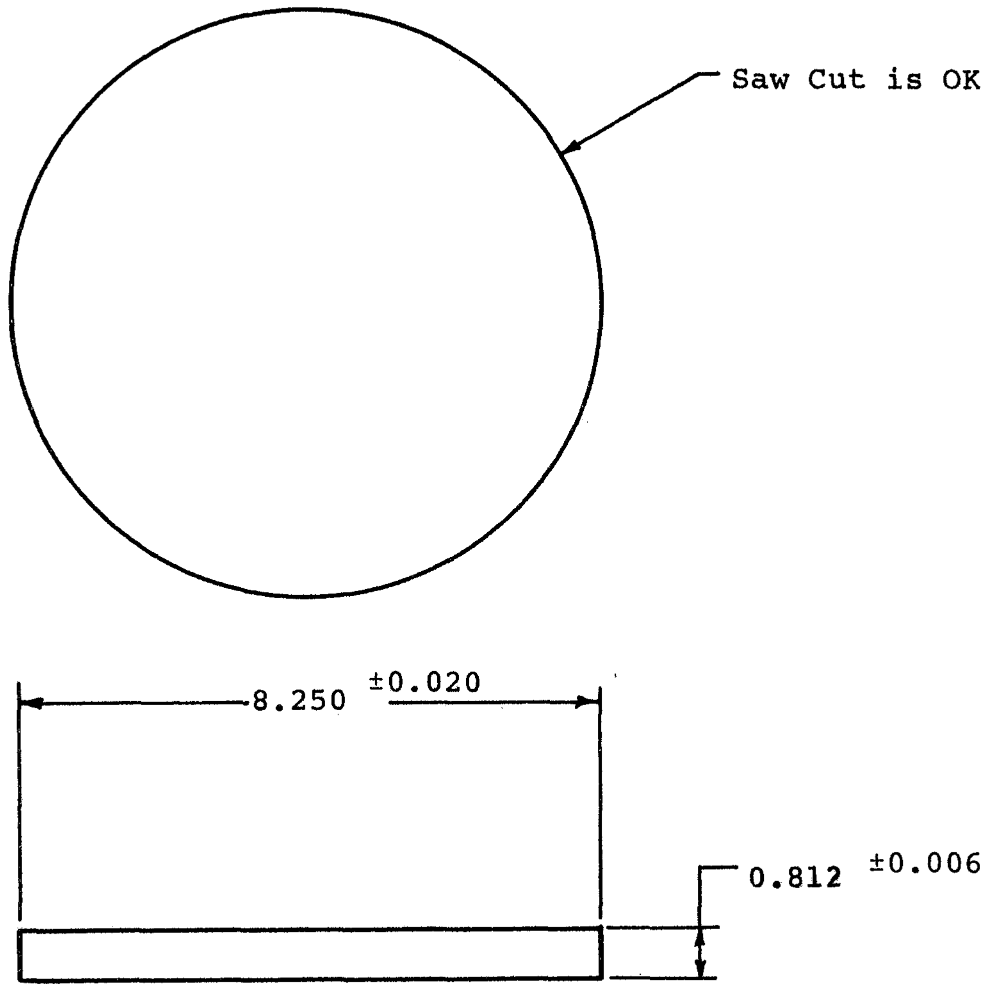
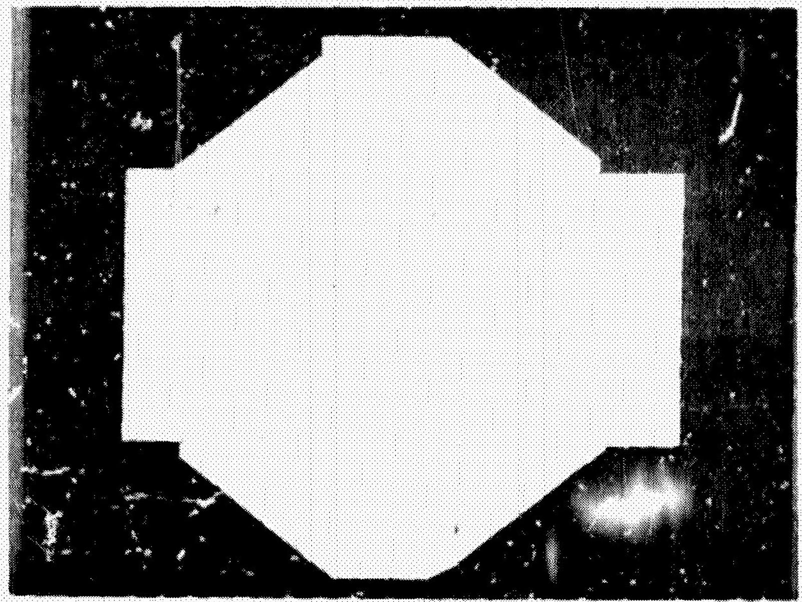
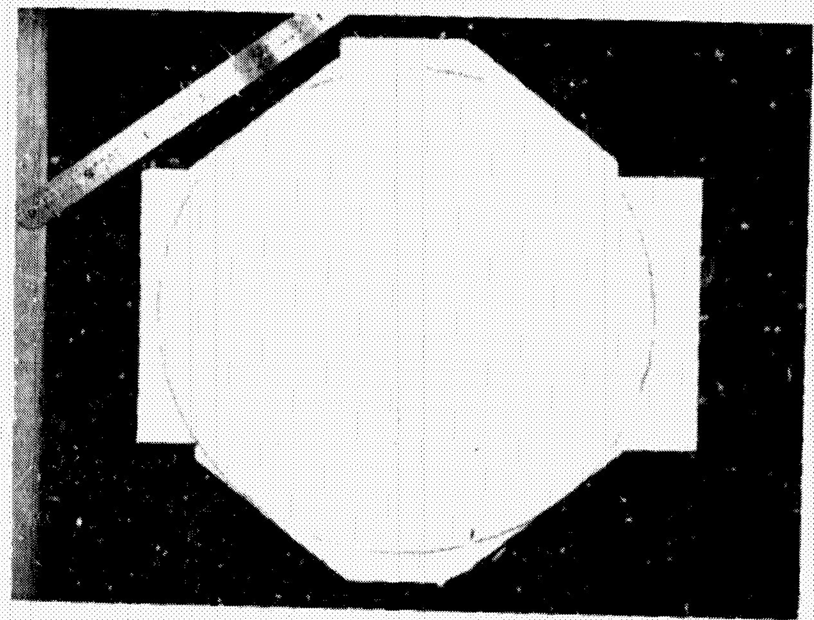


Figure 5. Thermal conductivity specimen for ASTM C177 thermal conductivity apparatus (3" diameter)



Arrangement of 0.635 cm thick pieces used to provide specimen size required



Picture showing cutting line for 18.40 cm diameter specimens

Figure 6. Pictures of thermal conductivity specimens for 7 inch ASTM guarded hot plate as made from 10.12 cm x 10.12 cm squares of Mullite Fiber Rigidized Insulation

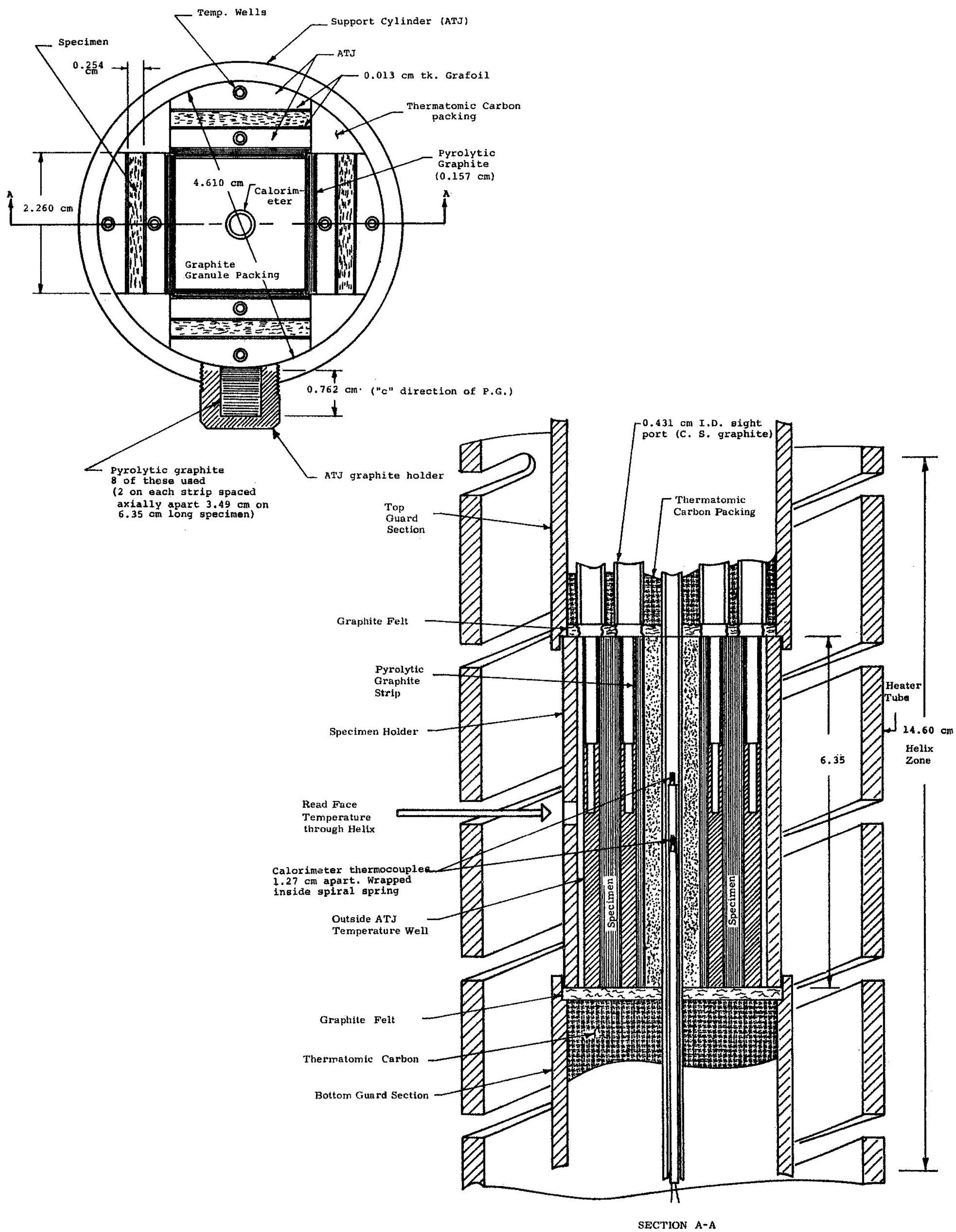
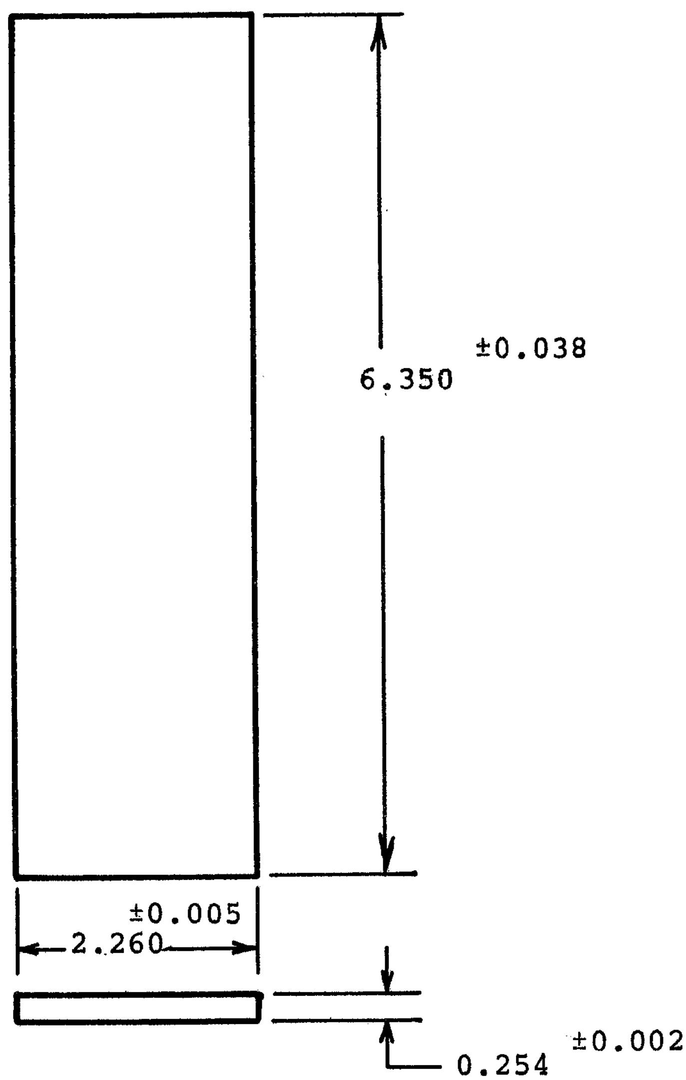
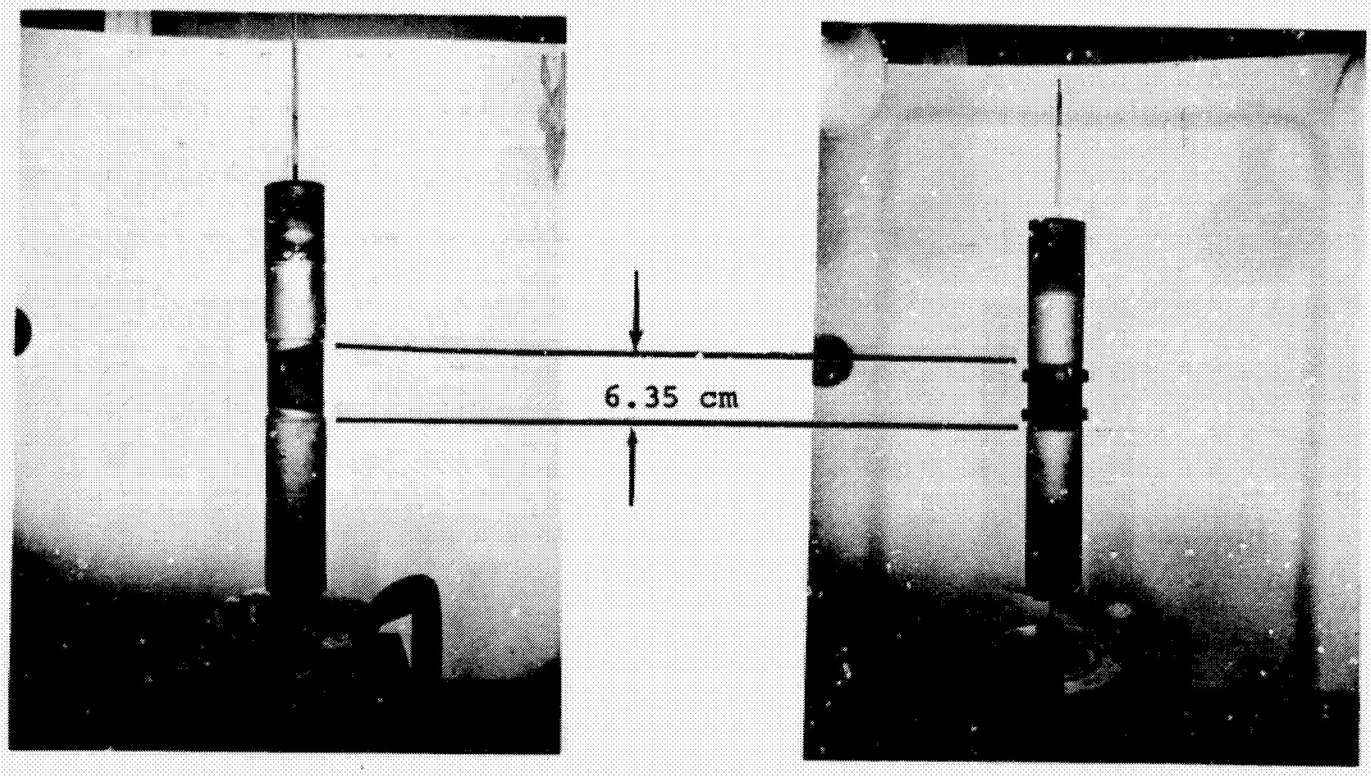


Figure 7. Schematic of strip specimen configuration for thermal conductivity measurements in radial inflow apparatus



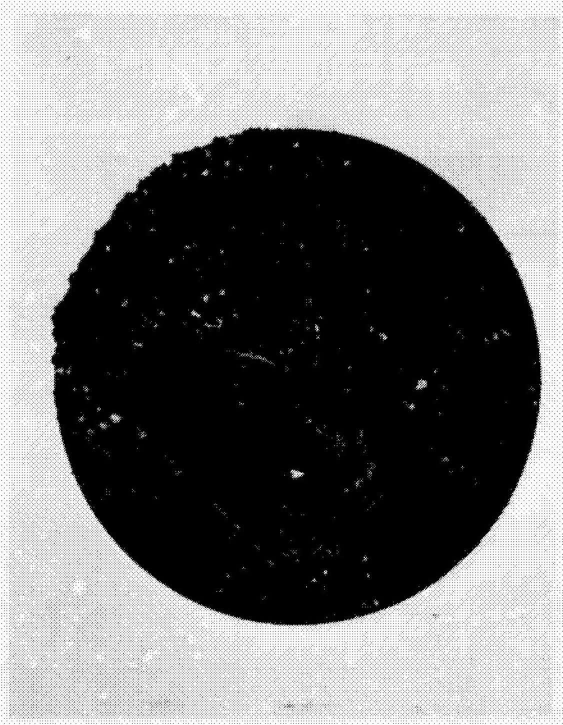
Notes: 1. Four strips comprise one specimen
2. Specimen AL-1 (alumina-silica-chromia) was 1.270 cm wide by 7.110 cm long and was evaluated under a positive compaction load

Figure 8. Configuration of specimen strips for radial inflow apparatus (RIA)

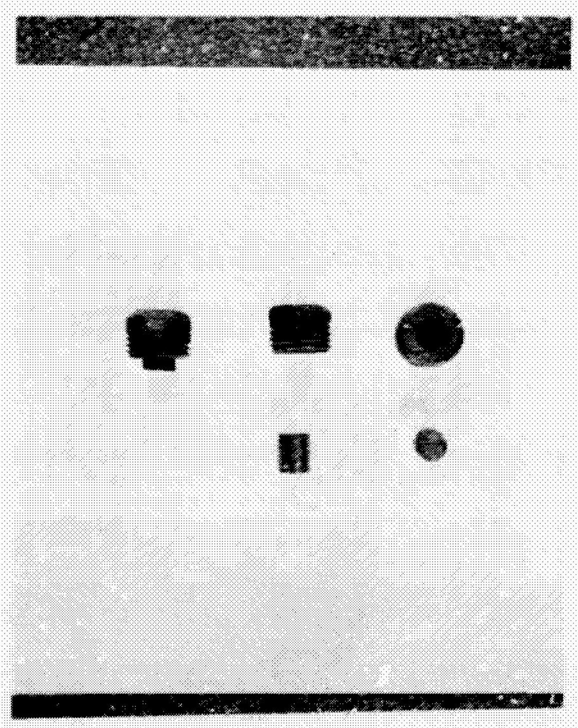


(a) Side View of Specimen Built Up on Calorimeter - Without PG Loading Pins

(b) Side View of Specimen Built Up on Calorimeter - With PG Loading Pins



(c) Top View of Specimen in Specimen Holder (RIA 1)



(d) PG Loading Pins and ATJ Cap

Figure 9. Pictures of apparatus for thermal conductivity measurements with radial inflow apparatus

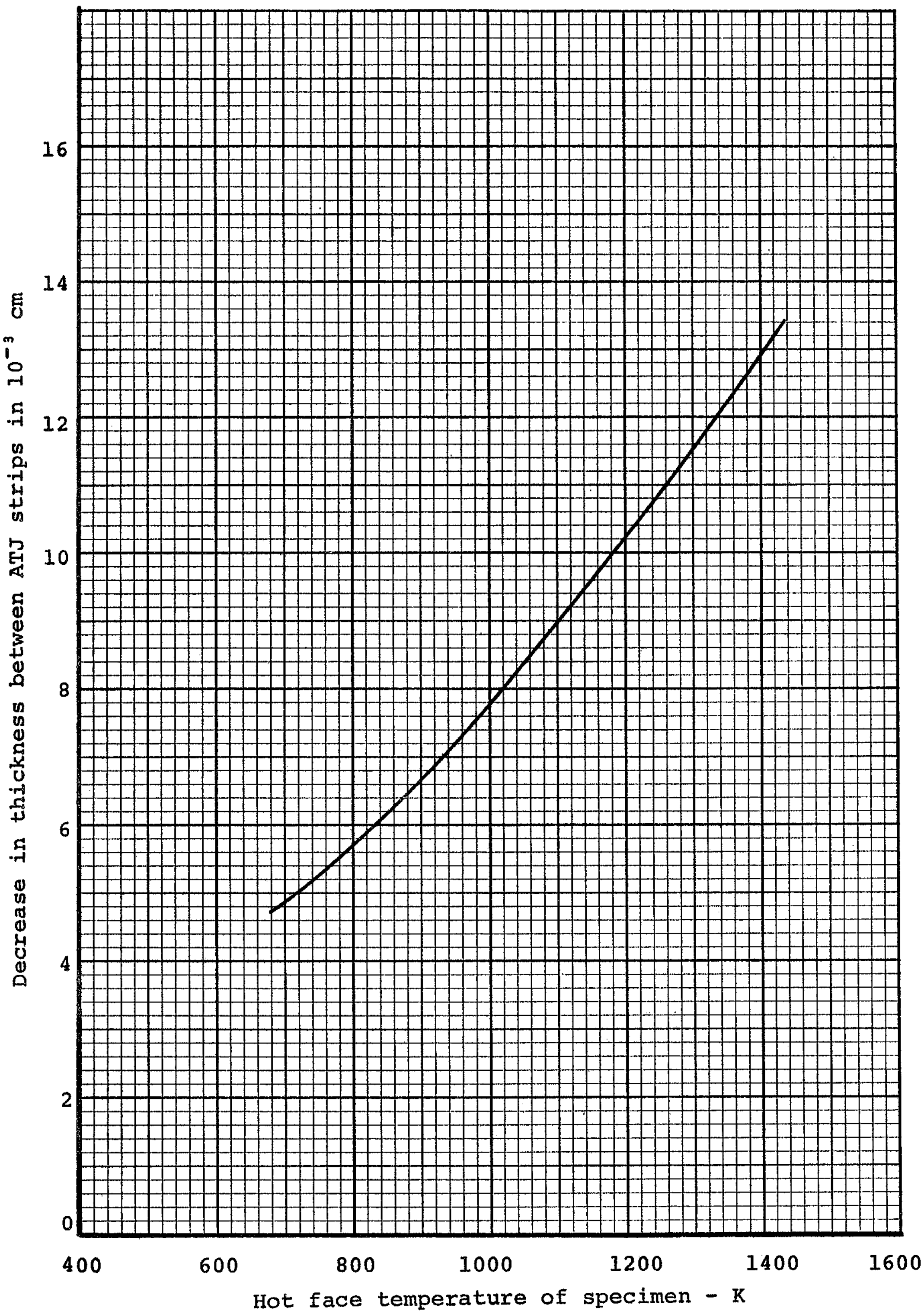


Figure 10. Thickness reduction imposed on thermal conductivity specimens during measurements with radial inflow apparatus in GFE furnace

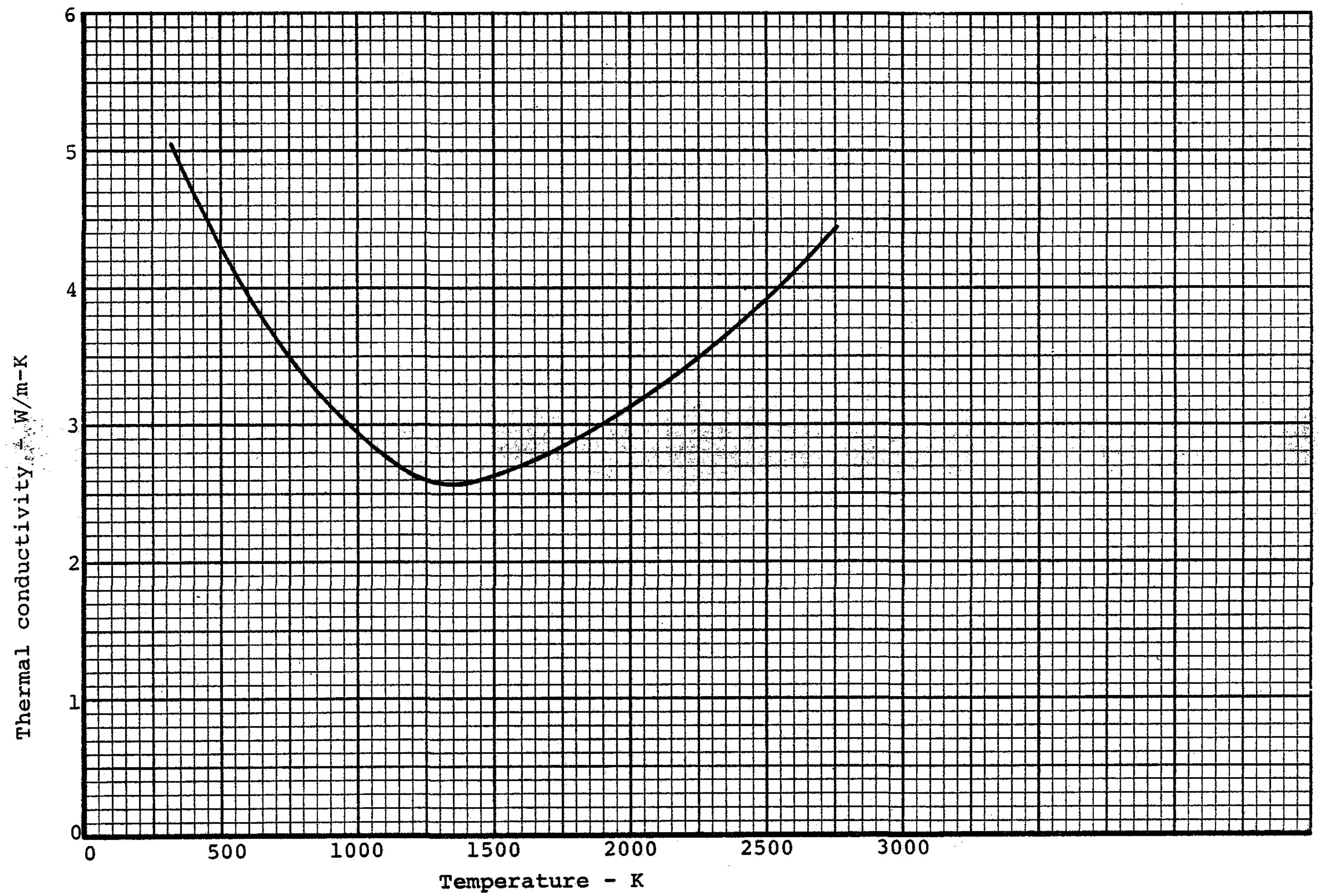


Figure 11. The thermal conductivity of plain Grafoil in the across lamina direction

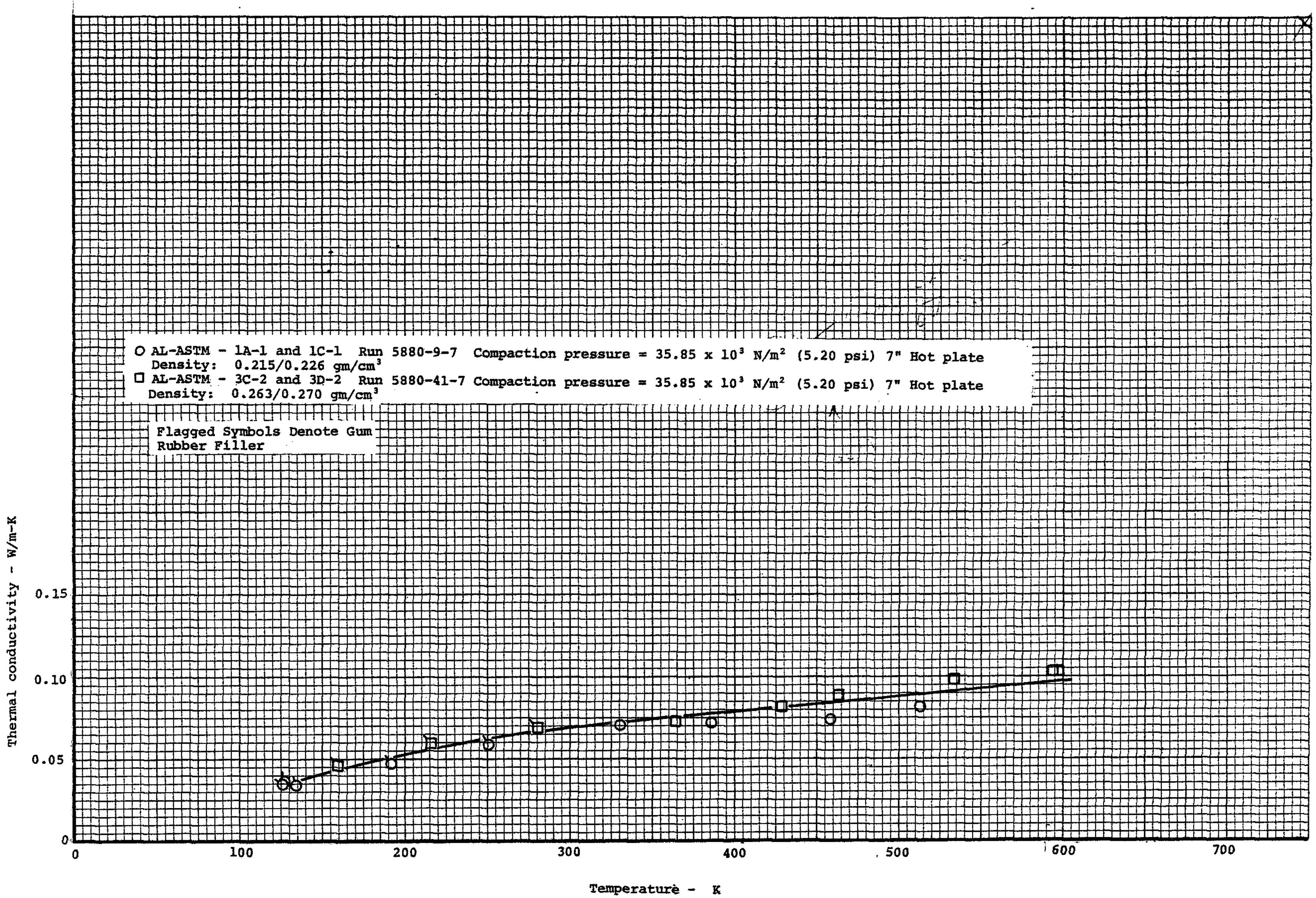


Figure 12. Thermal conductivity of Alumina-Silica-Chromia Rigidized Insulation in air in the across lamina direction - ASTM C177 guarded hot plate apparatus

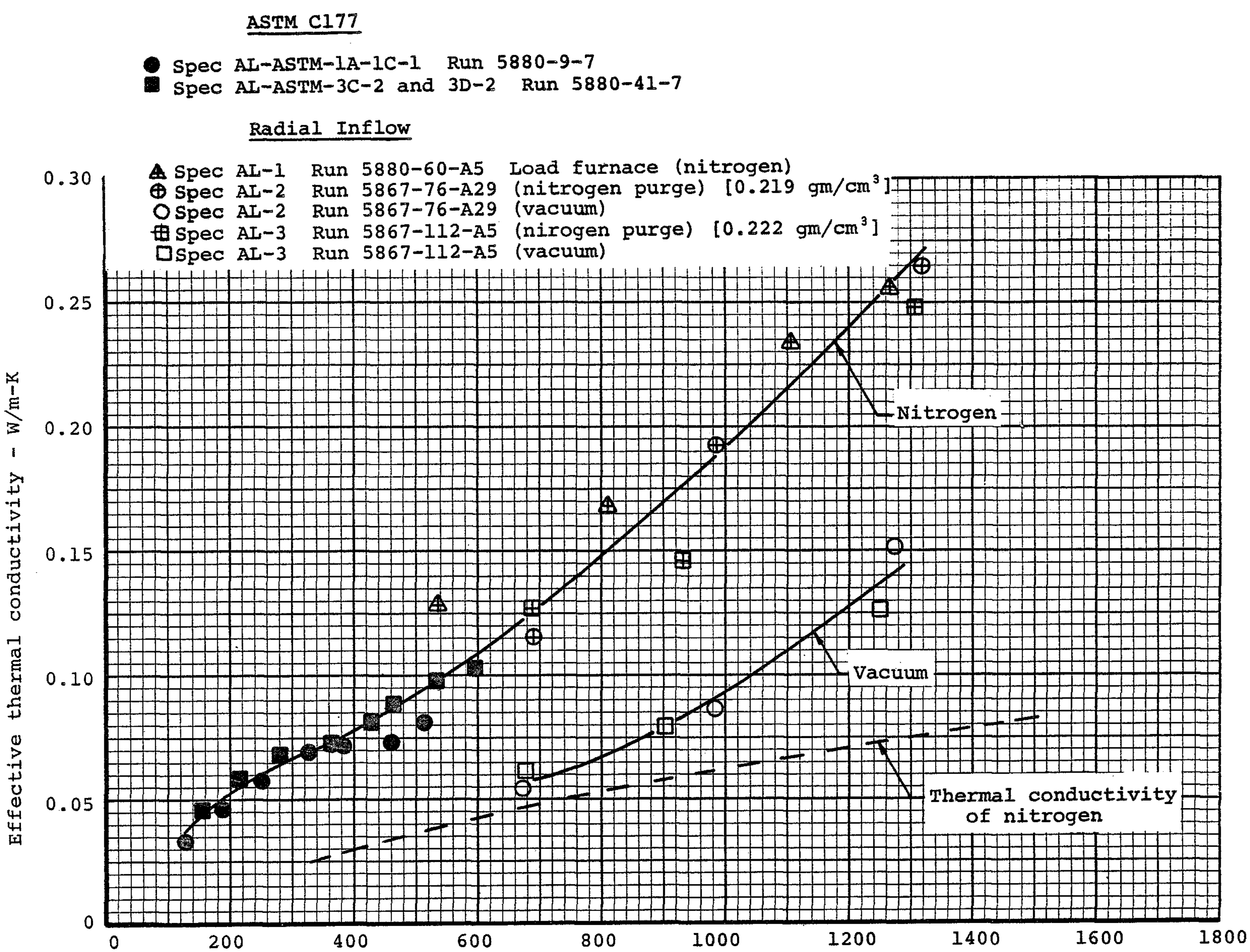


Figure 13. Effective thermal conductivity of Alumina-Silica-Chromia Rigidized Insulation in nitrogen and vacuum

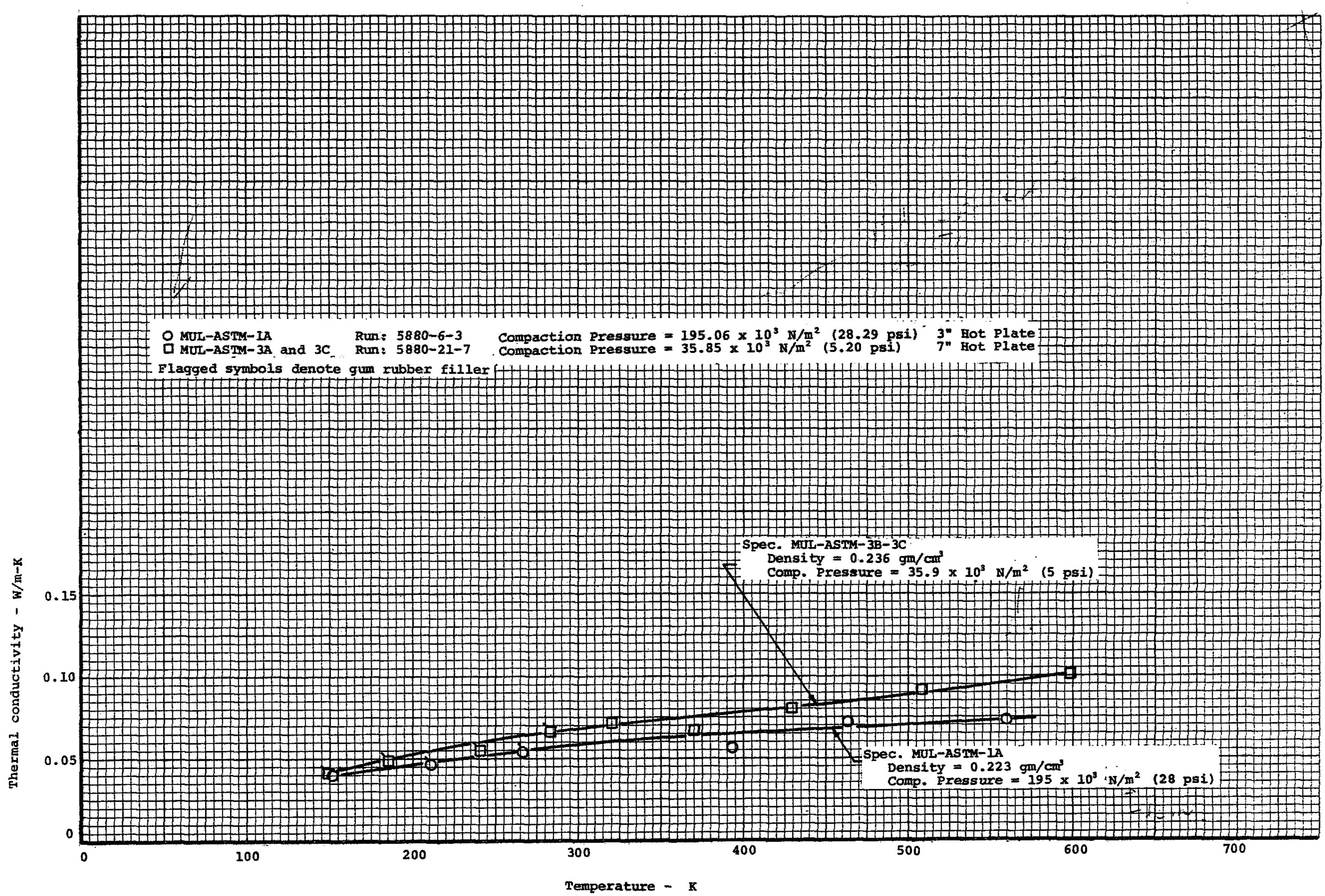


Figure 14. Thermal conductivity of Mullite Fiber Rigidized Insulation in air in the across lamina direction - ASTM C177 guarded hot plate apparatus

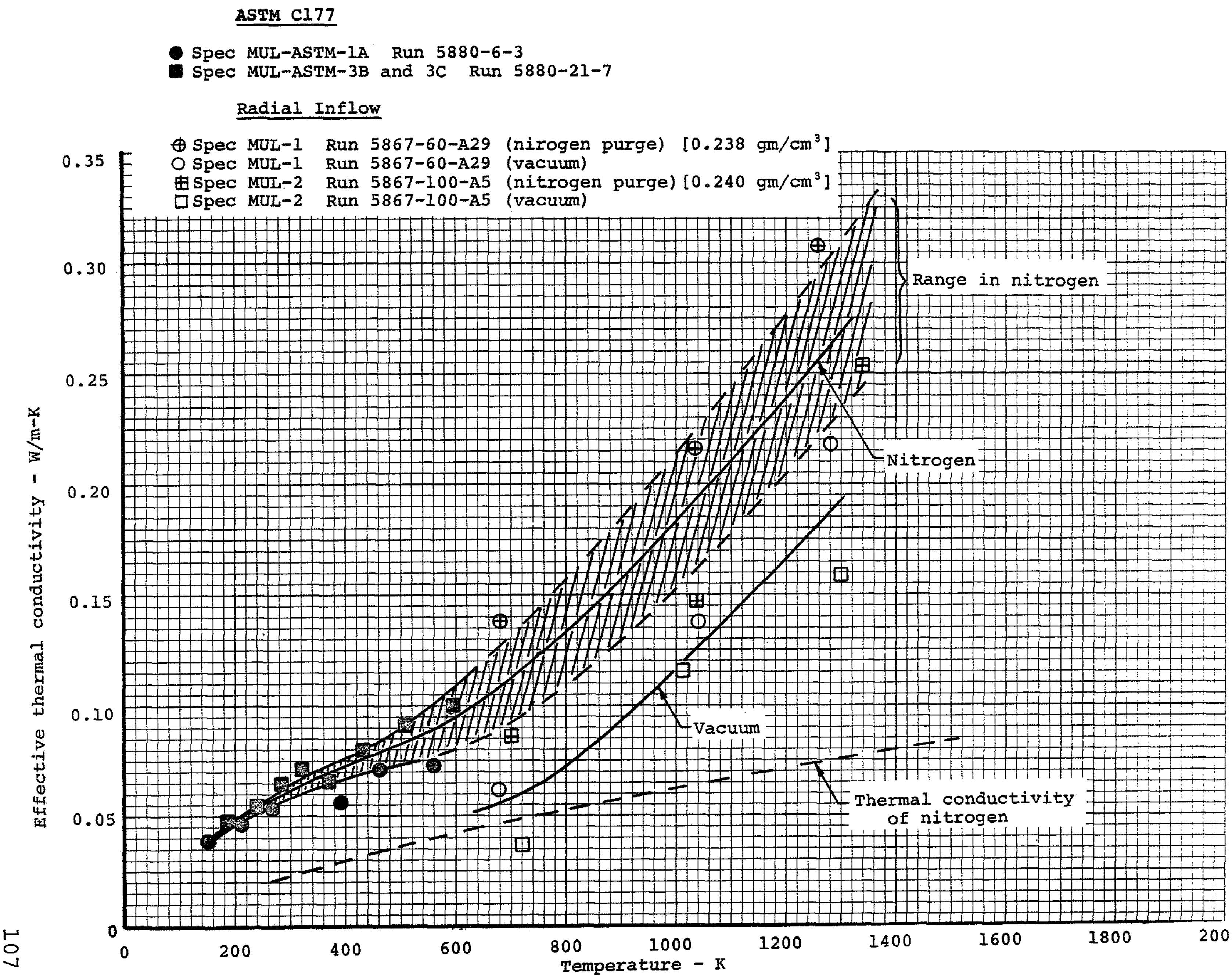


Figure 15. Effective thermal conductivity of Mullite Fiber Rigidized Insulation in nitrogen and vacuum

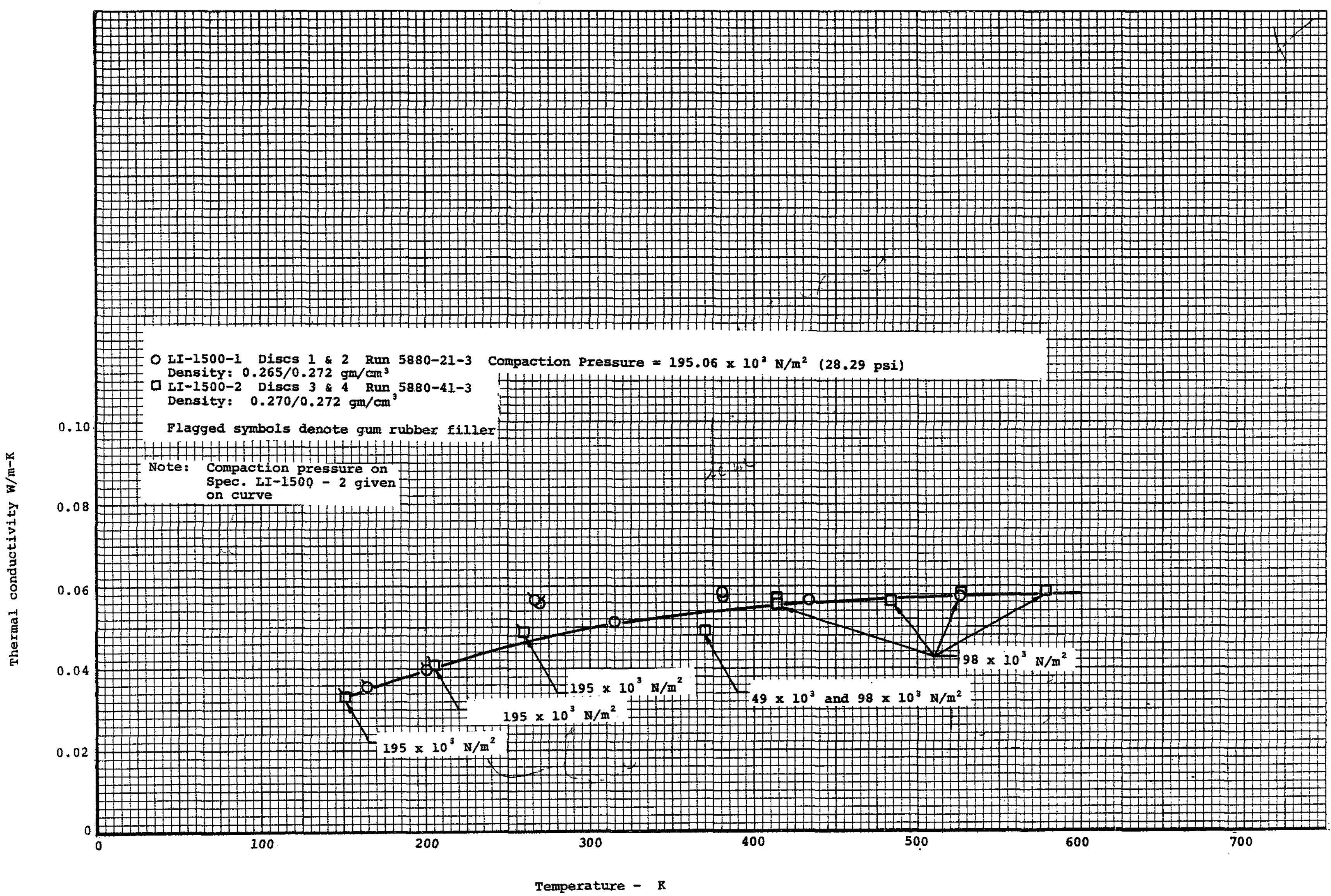


Figure 16. Thermal conductivity of LI-1500 Rigidized Insulation in air in the across lamina direction - ASTM C177
3 inches diameter guarded hot plate

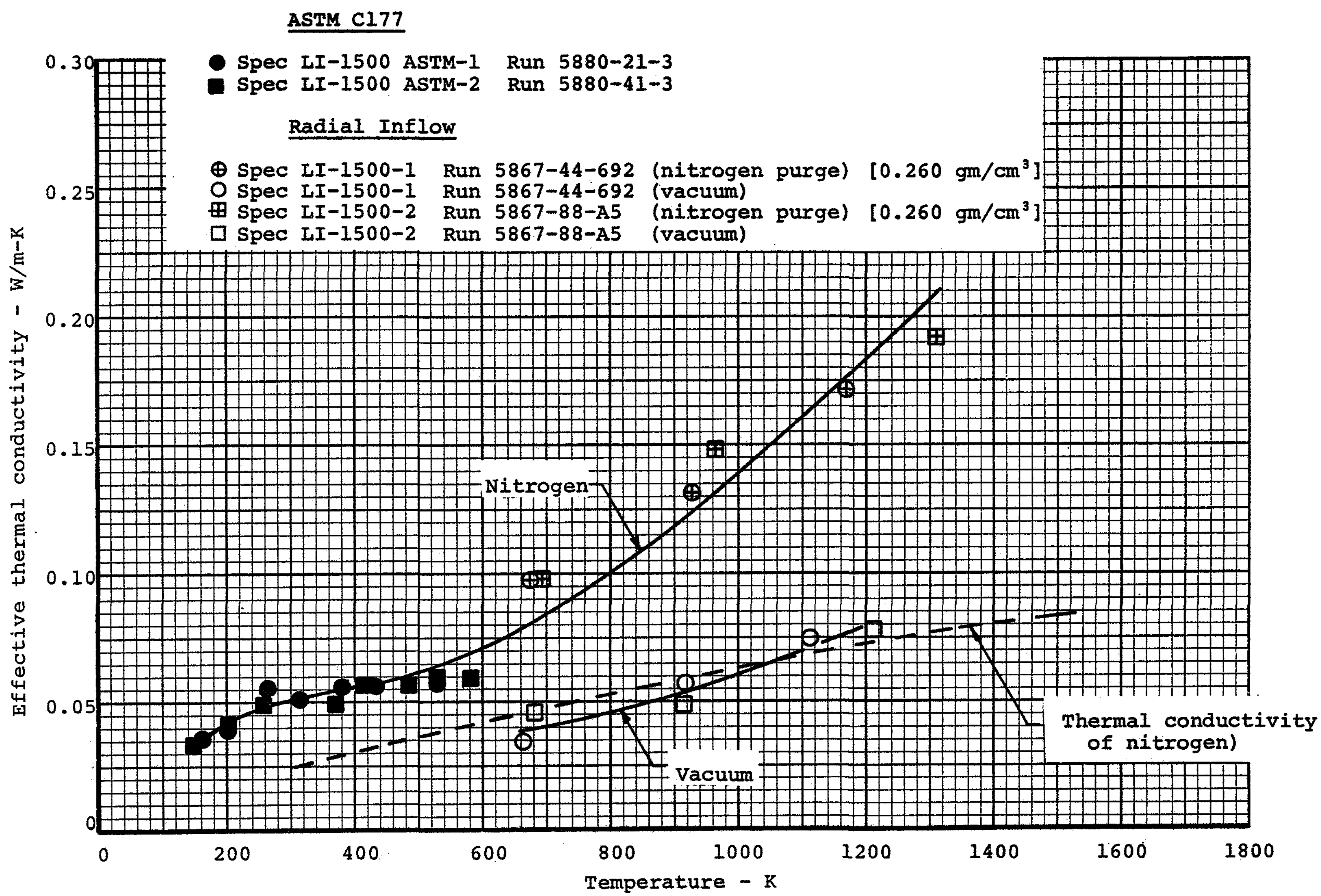


Figure 17. Effective thermal conductivity of LI-1500 in nitrogen and vacuum

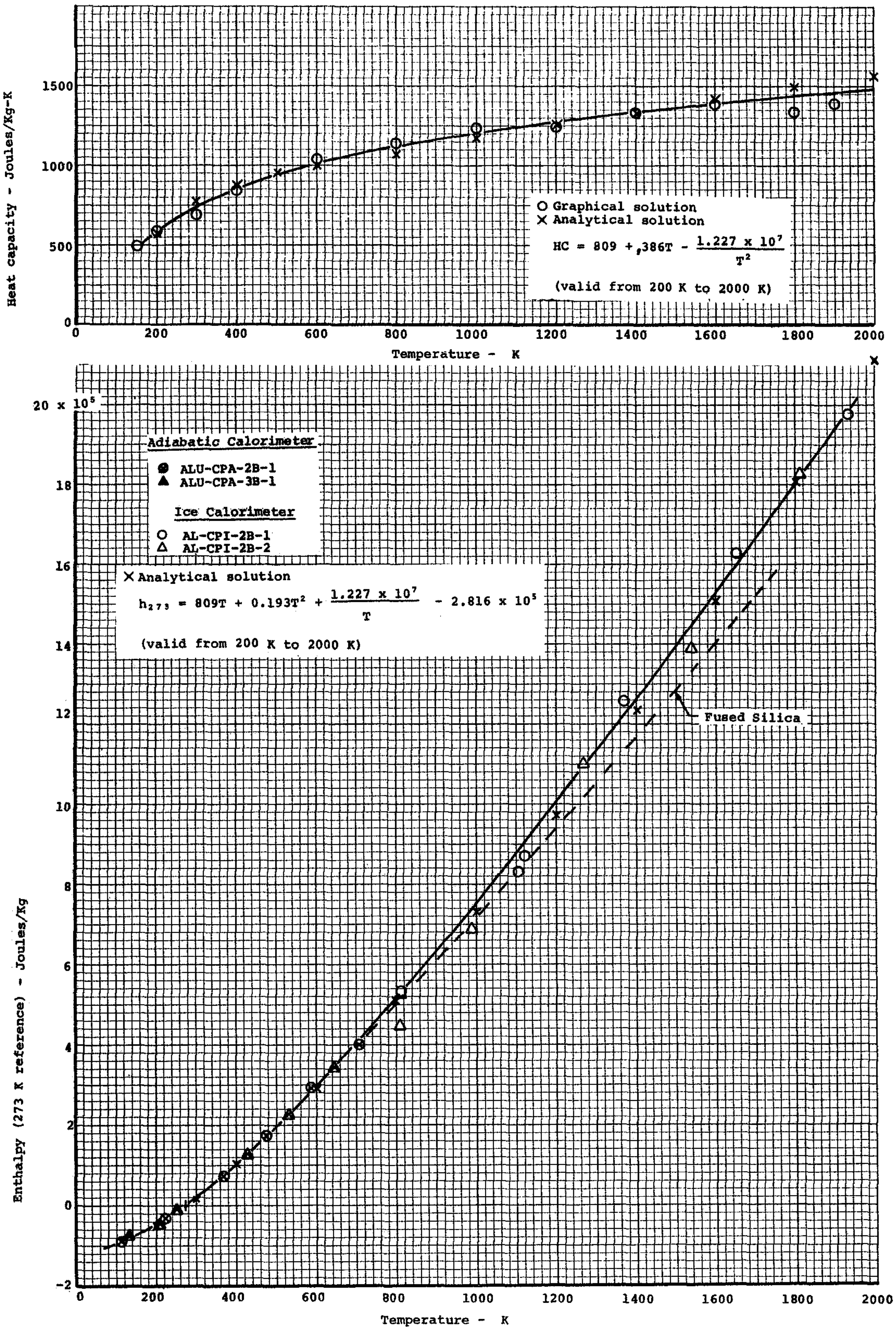
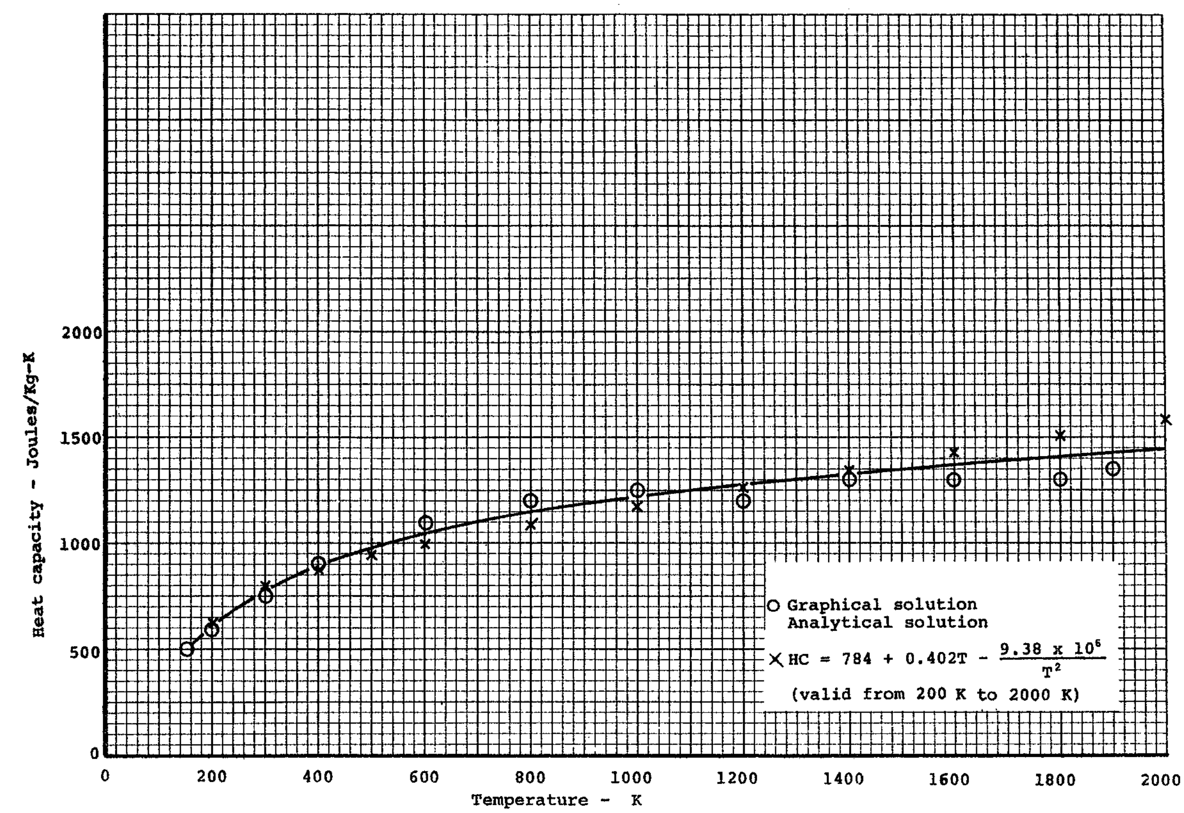


Figure 18. Enthalpy and heat capacity of Alumina-Silica-Chromia Rigidized Insulation



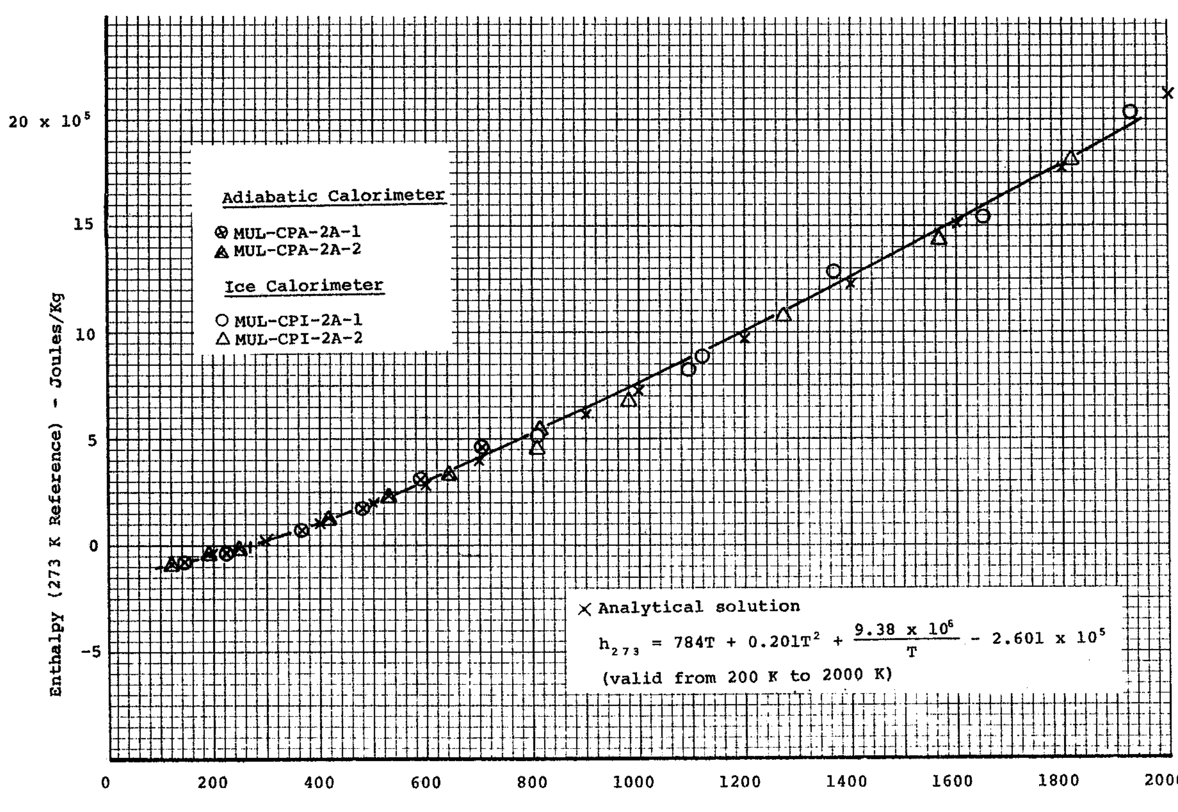


Figure 19. Enthalpy and heat capacity of Mullite Fiber Rigidized Insulation

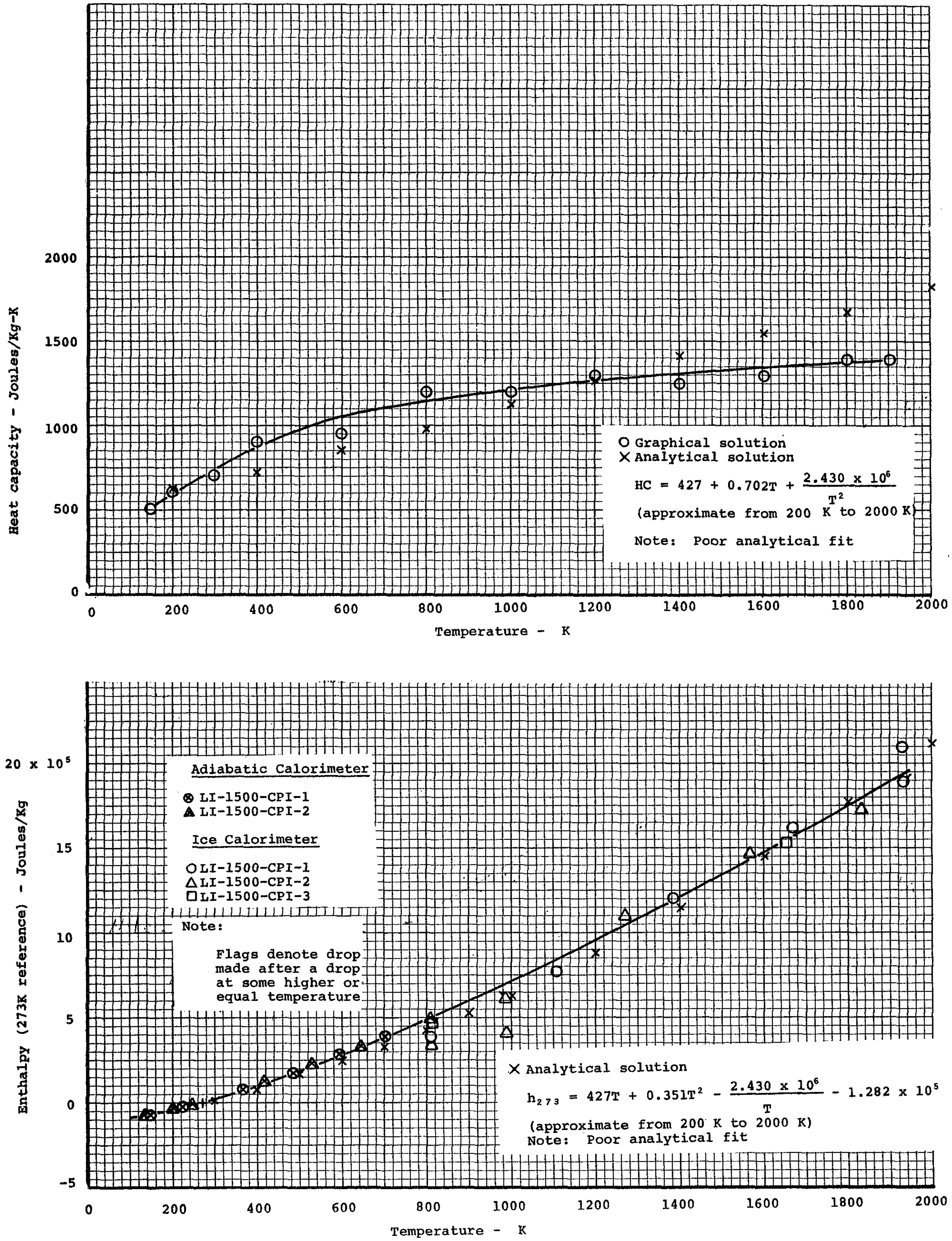
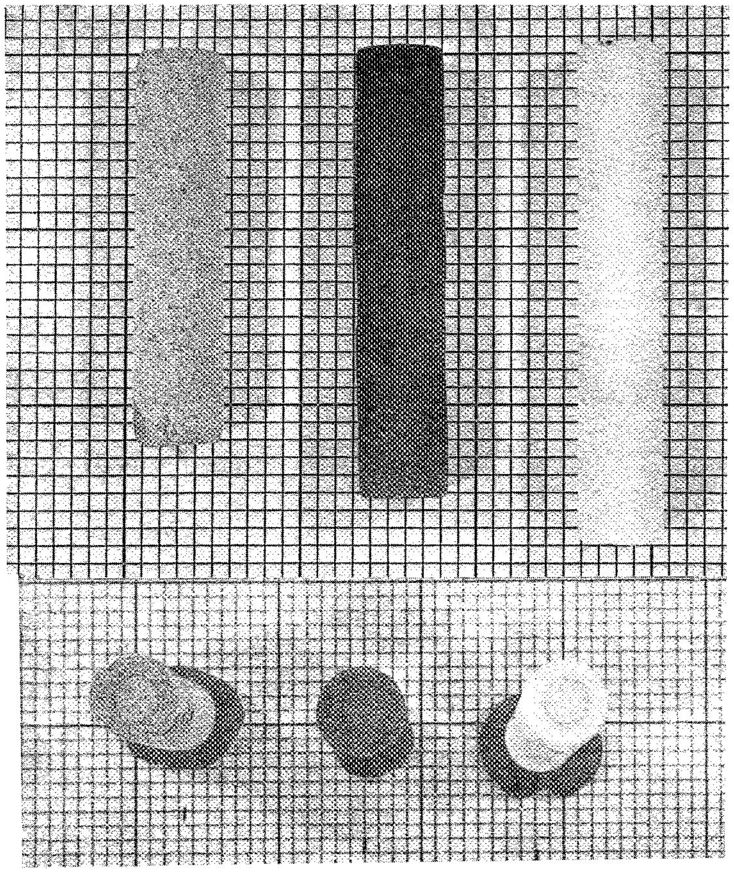
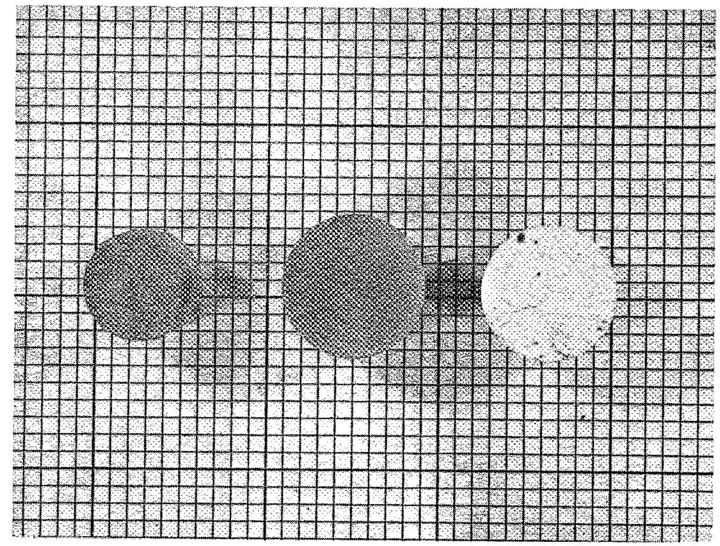


Figure 20. Enthalpy and heat capacity of LI-1500 Rigidized Insulation



(Al-Si-Cr)	(Mullite)	(LI-1500)
AL-GEXP-1B-W	MUL-GEXP-1CW	LI-1500-GEXP-W2
Exposed to 1497K	Exposed to 1722K	Exposed to 1883K

(a) Thermal Expansion Specimens



(Al-Si-Cr)	(Mullite)	(LI-1500)
AL-CPI-2B-2	MUL-CPI-2A-2	LI-1500-CPI-1
Exposed to 1811K	Exposed to 1819K	Exposed to 1928K

Note: These specimens were initially powdered

(b) Enthalpy Specimens

Figure 21. Pictures of thermal expansion and enthalpy specimens - post exposure

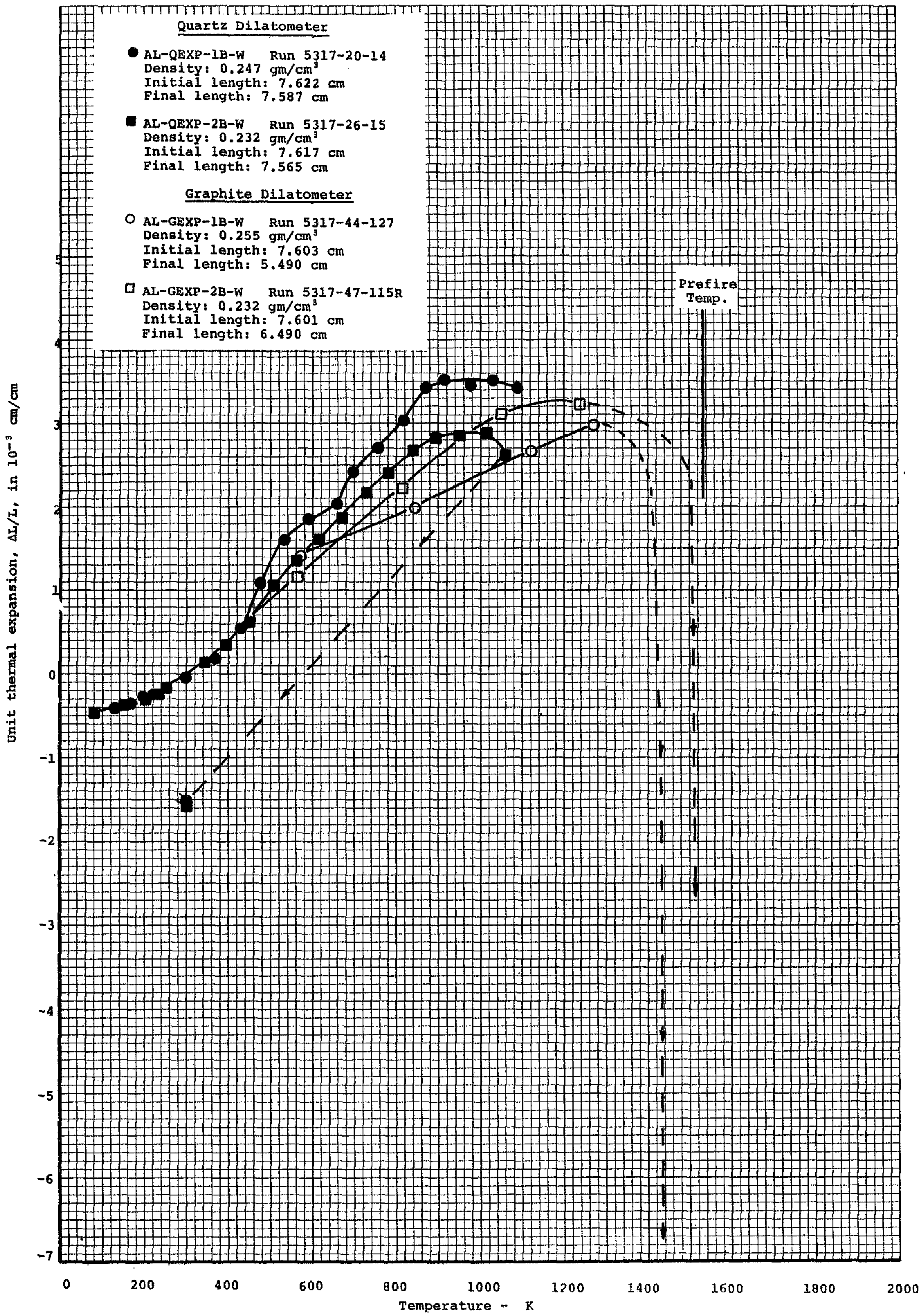


Figure 22. Thermal expansion of Alumina-Silica-Chromia Rigidized Insulation in the with lamina direction

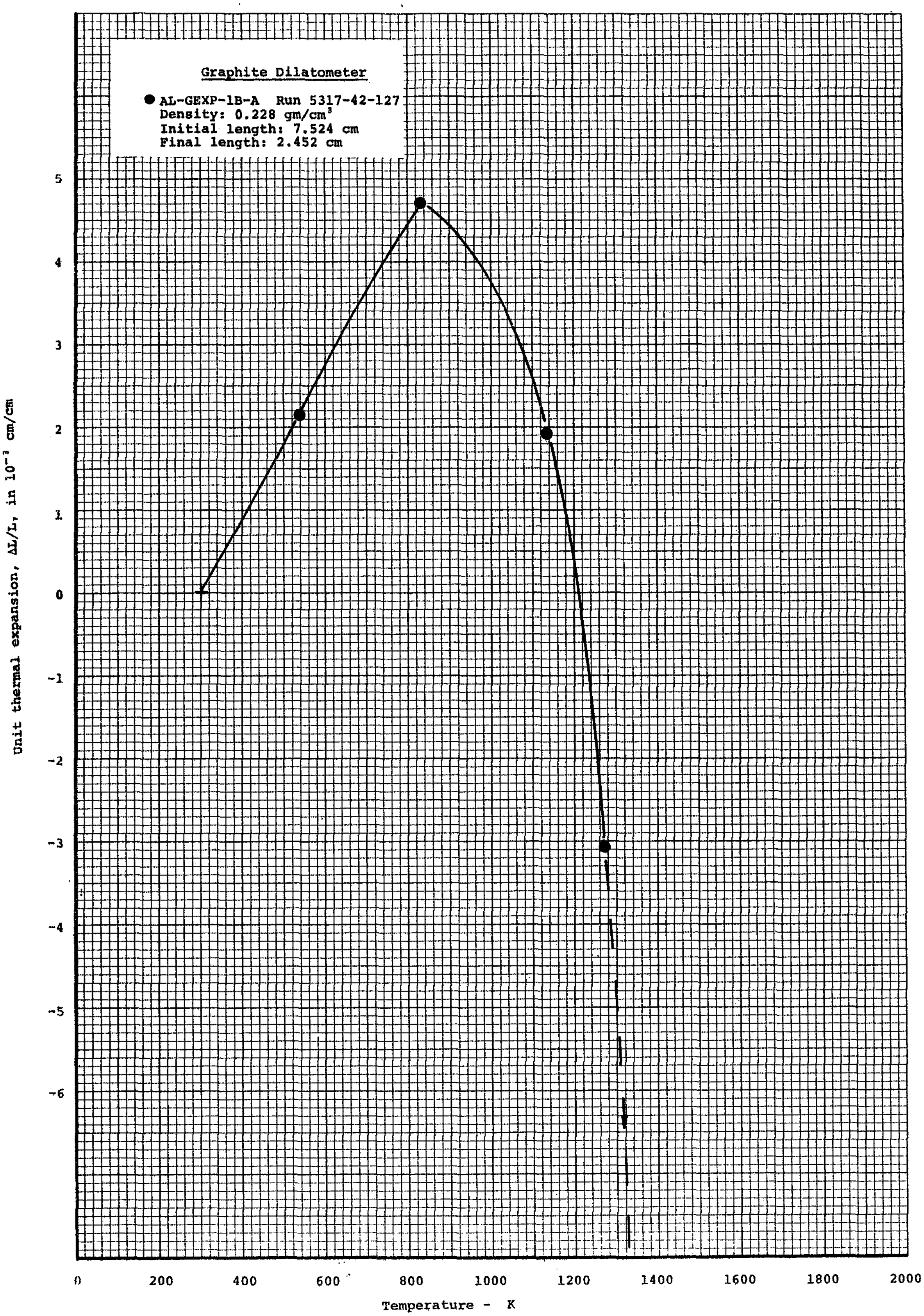


Figure 23. Thermal expansion of Alumina-Silica-Chromia Rigidized Insulation in the across lamina direction

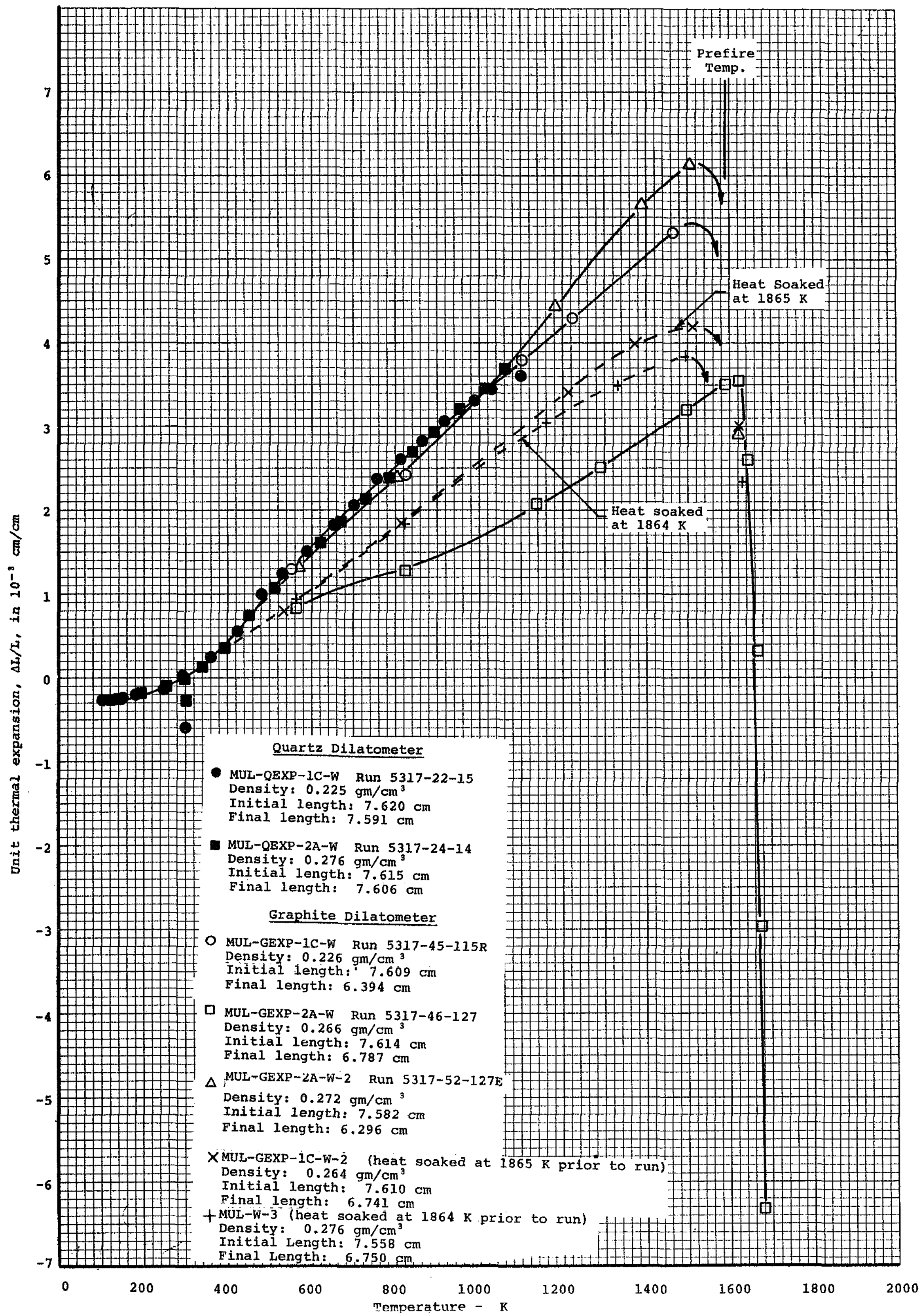


Figure 24. Thermal expansion of Mullite Fiber Rigidized Insulation in the with lamina direction

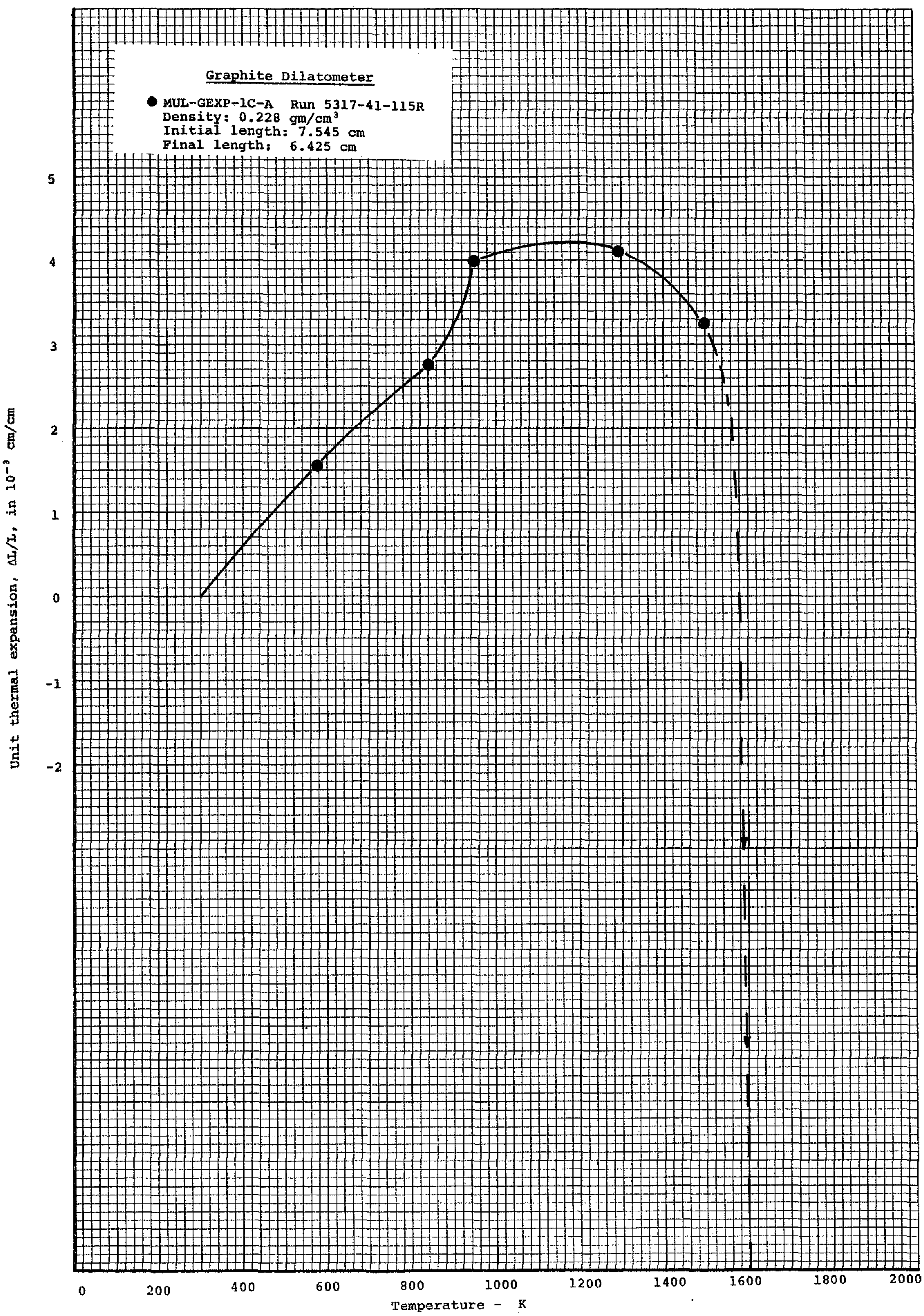


Figure 25. Thermal expansion of Mullite Fiber Rigidized Insulation in the across lamina direction

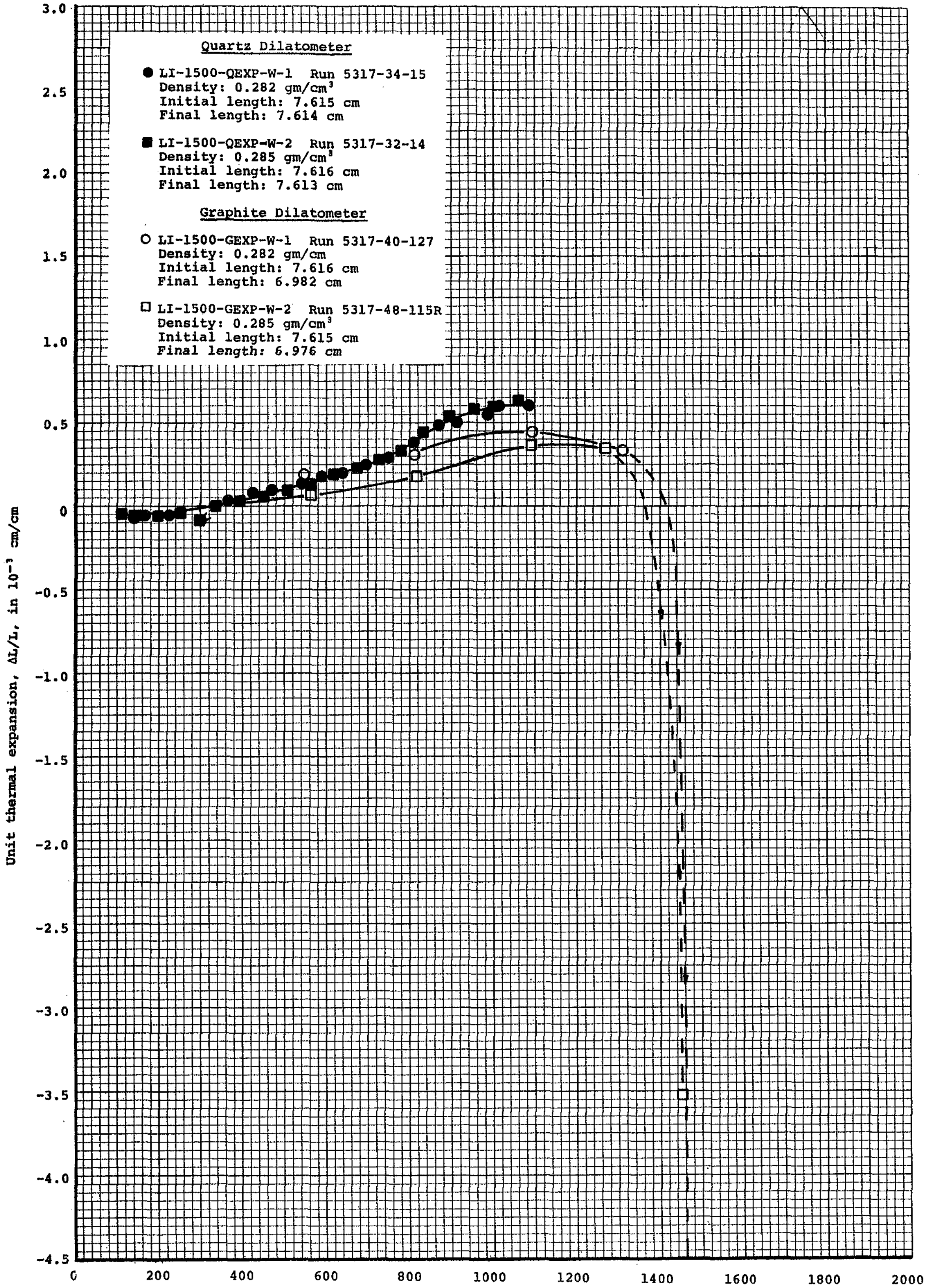


Figure 26. Thermal expansion of LI-1500 Rigidized Insulation in the with lamina direction

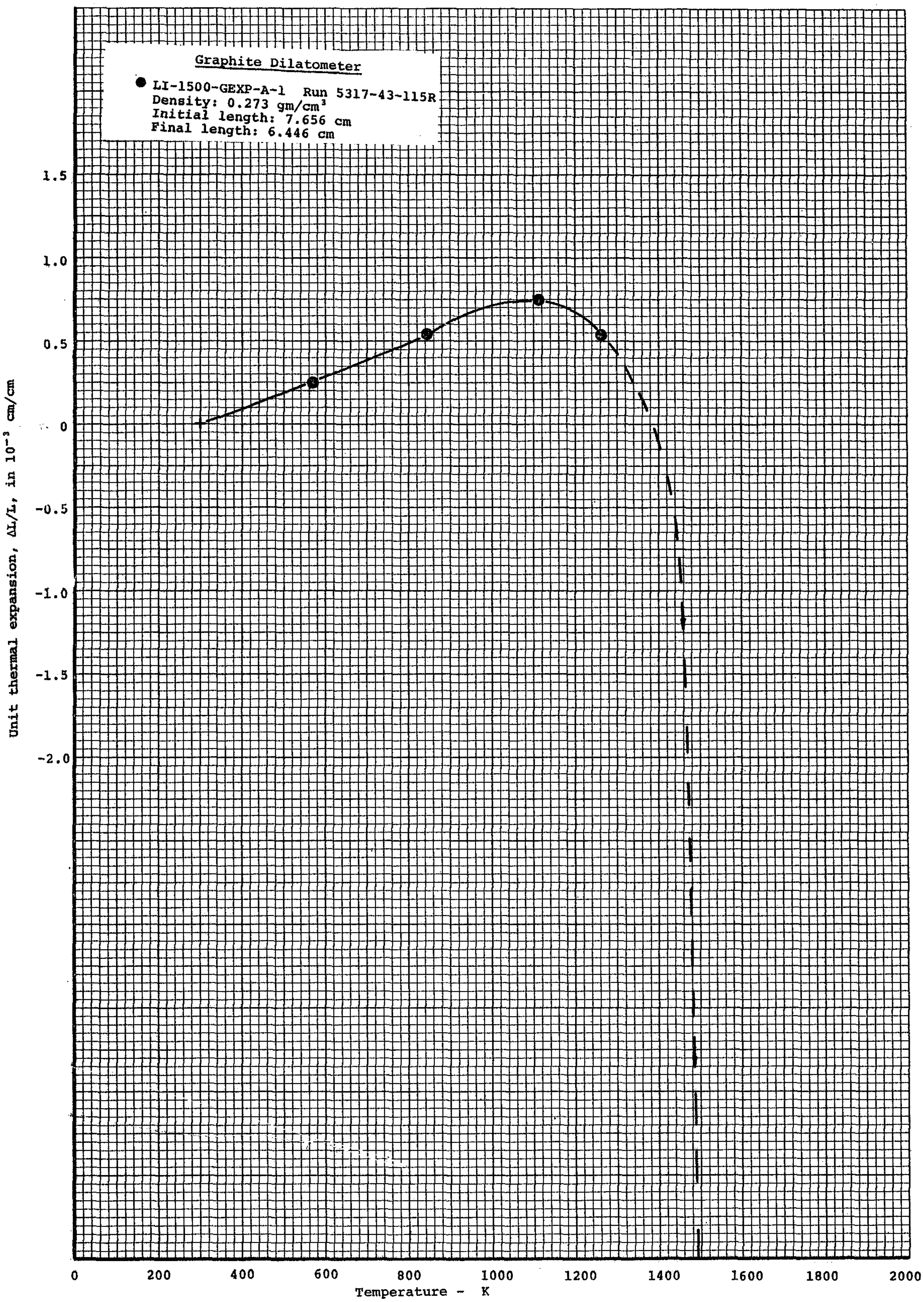


Figure 27. Thermal expansion of LI-1500 Rigidized Insulation in the across lamina direction

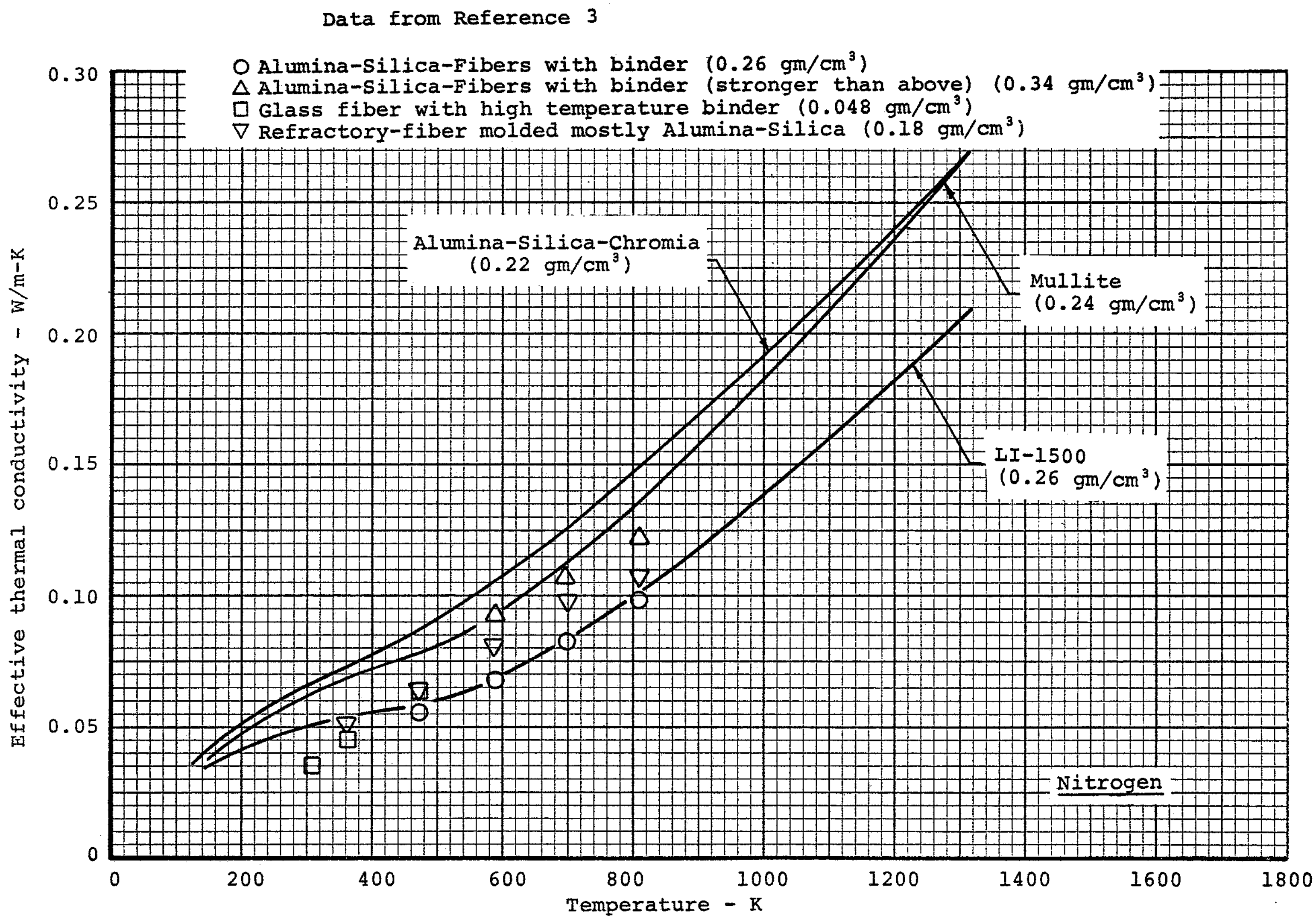


Figure 28. Effective thermal conductivity of three rigidized insulators in a nitrogen environment

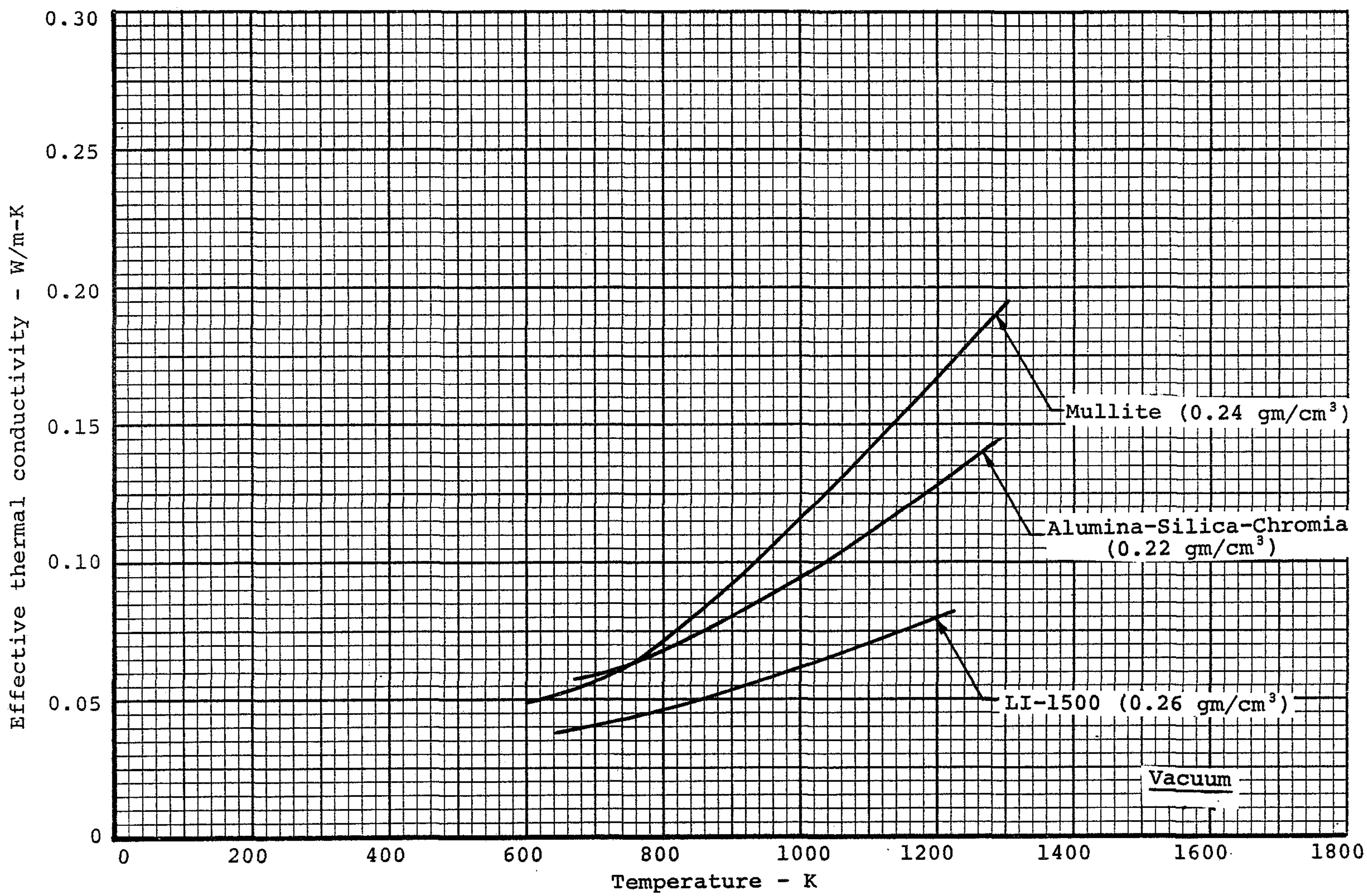


Figure 29. Effective thermal conductivity of three rigidized insulators in a vacuum environment

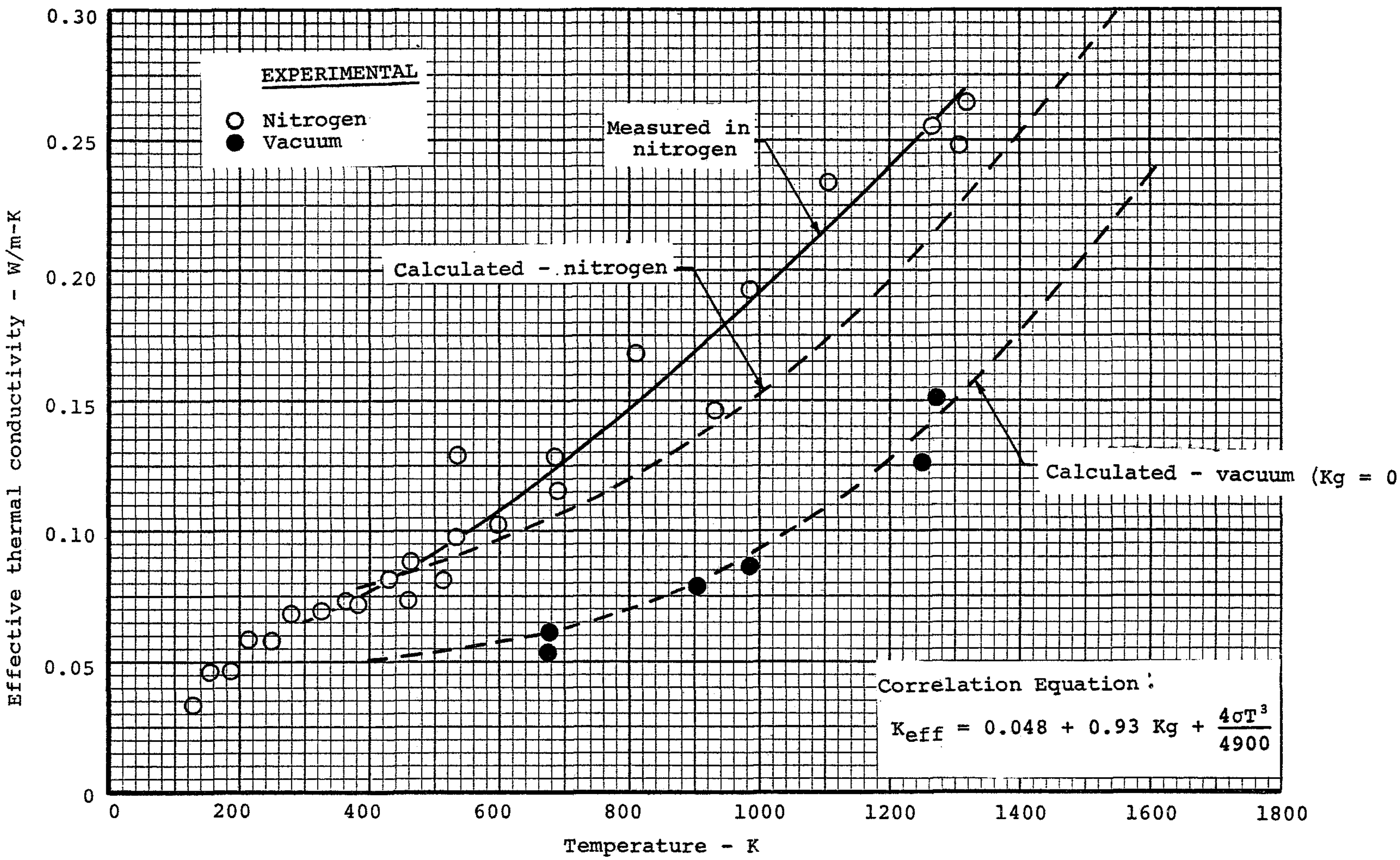


Figure 30. Analytical correlation of data for Alumina-Silica-Chromia Rigidized Insulation

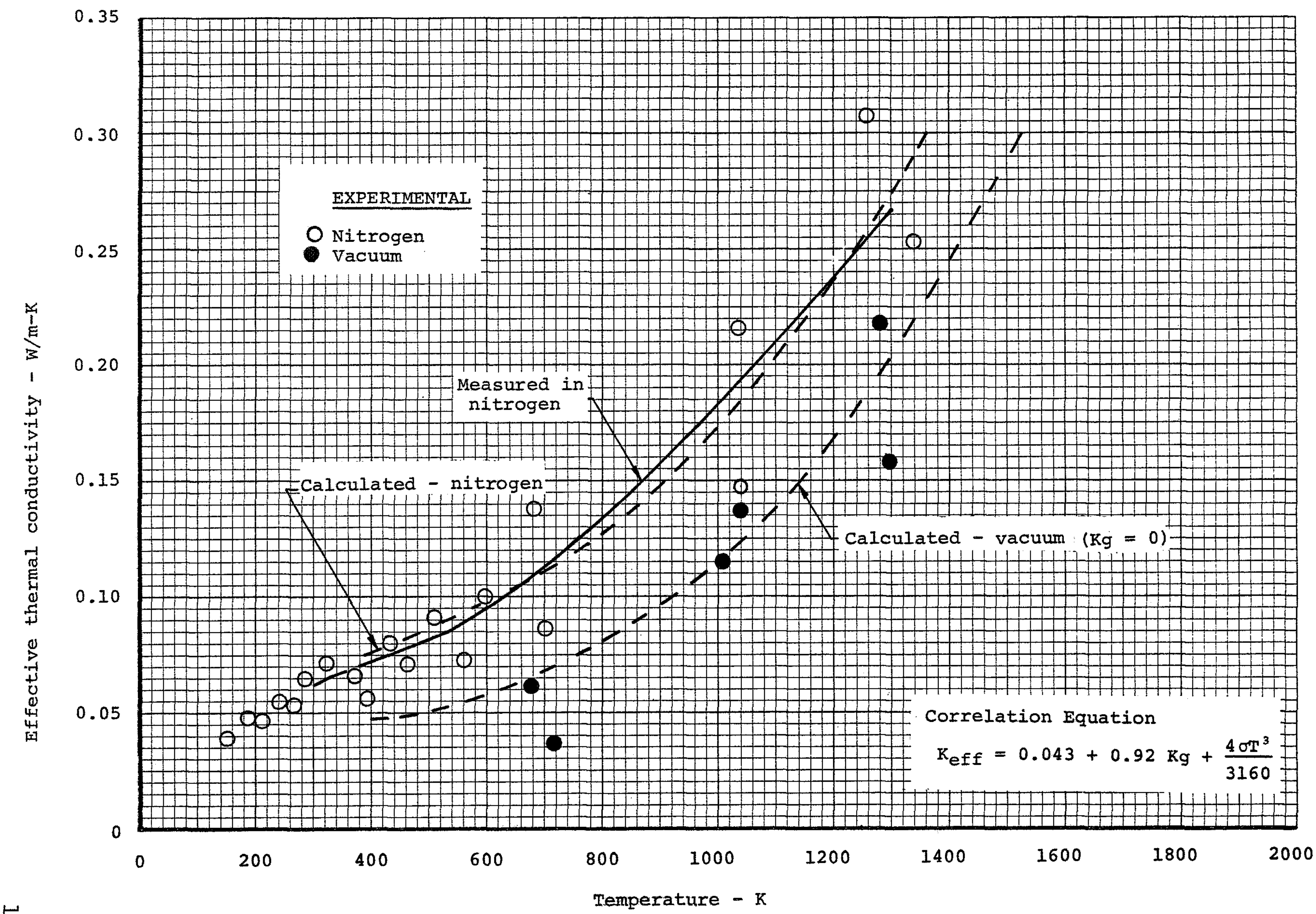


Figure 31. Analytical correlation of data for Mullite Fiber Rigidized Insulation

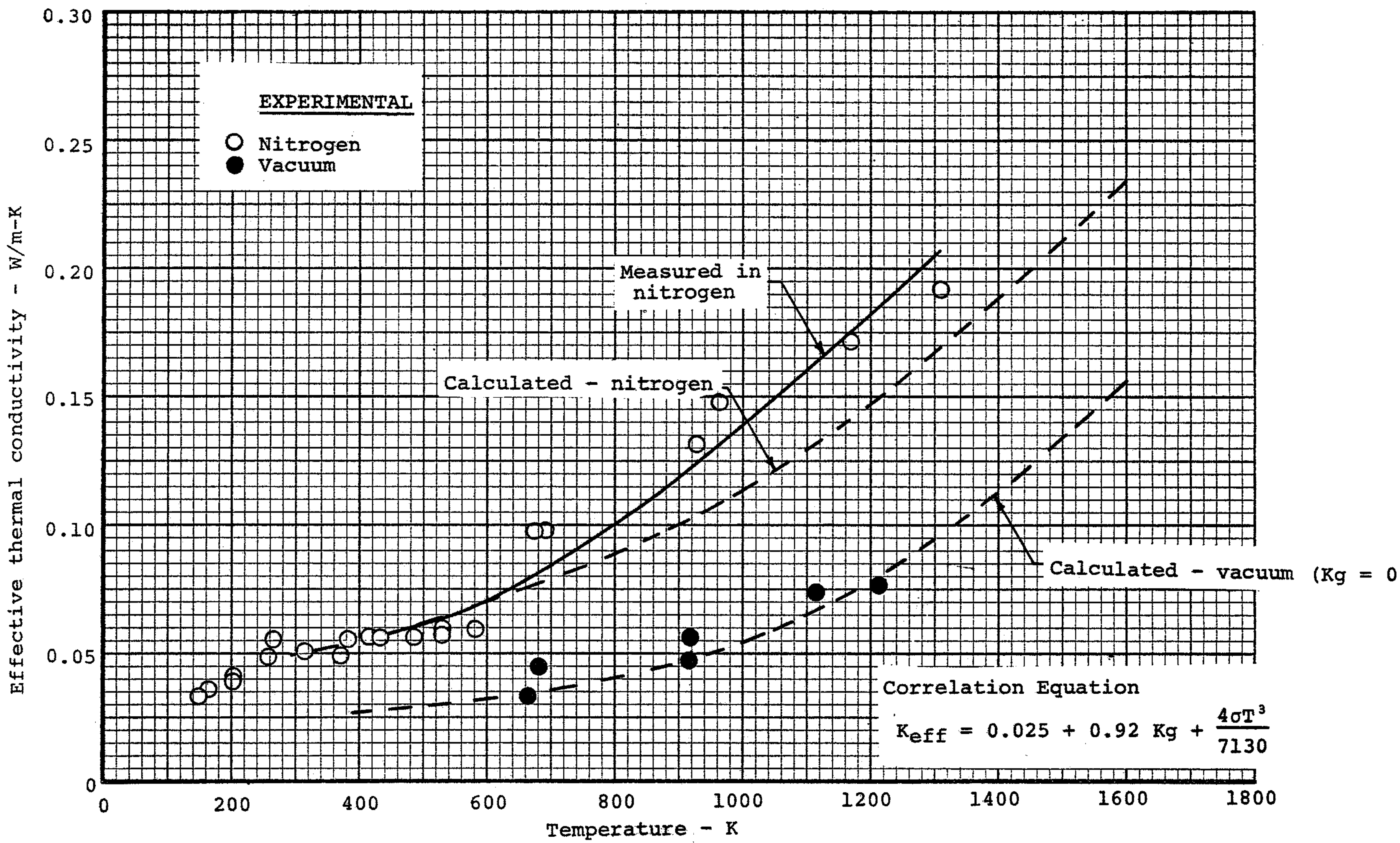


Figure 32. Analytical correlation of data for LI-1500 Rigidized Insulation

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